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THESIS

THE ANALYSIS OF RANDOM EFFECTS REGRESSION MODEL FOR PREDICTING THE SHELF-LIFE OF GUN PROPELLANT

by

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Most gun propellant is stored at depots for a long time before it is used. While being stored, the quality of the gun propellant may deteriorate and become unstable. In an attempt to avoid disaster due to use of unstable gun propellant, accurate prediction of the safe shelf-life of gun propellant is necessary. The shelf-life estimation methods used currently for a group of similar gun propellant lots are based on a fixed effects regression model. This does not take into consideration the fact that samples from the same lot are more similar than samples between lots. To capitalize on this lot-to-lot variation when estimating the shelf-life, first, a random effects regression model is developed. Secondly, a combined mixed effects model is estimated. The estimated model is then used to predict not only the shelf-life of a group of similar lots but also that of each individual lot of 5"/54 NACO gun propellant stockpile. The results indicate that, first, the claimed shelf-life is not adequate and requires amendment. Next, the group shelf-life estimated can be relatively conservative compared to the individual shelf-lives. In view of potential opportunity loss due to safe individual lots being discarded. use of individual shelf-life is recommended.

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ABSTRACT

Most gun propellant is stored at depots for a long time before it is used. While being stored, the quality of the gun propellant may deteriorate and become unstable. In an attempt to avoid disaster due to use of unstable gun propellant, accurate prediction of the safe shelf-life of gun propellant is necessary. The shelf-life estimation methods used currently for a group of similar gun propellant lots are based on a fixed effects regression model. This does not take into consideration the fact that samples from the same lot are more similar than samples between lots. To capitalize on this lot-to-lot variation when estimating the shelf-life, first, a random effects regression model is developed. Secondly, a combined mixed effects model is estimated. The estimated model is then used to predict not only the shelf-life of a group of similar lots but also that of each individual lot of 5"/54 NACO gun propellant stockpile. The results indicate that, first, the claimed shelf-life is not adequate and requires amendment. Next, the group shelf-life estimated can be relatively conservative compared to the individual shelf-lives. In view of potential opportunity loss due to safe individual lots being discarded, use of individual shelf-life is recommended.

TABLE OF CONTENTS

EXECUTIVE SUMMARY

Most gun propellant is stored at depots for a long time before it is used. While being stored, the quality of the gun propellant may deteriorate and become unstable. In an attempt to avoid disaster due to use of unstable gun propellant, accurate prediction of the safe shelf-life of gun propellant is necessary. Before 1914, real time storage inspection was used to estimate the safe shelf-life of gun propellant lots produced in the U.S.A. In an effort to reduce the length of time required for real time storage inspection, an Accelerated Aging Test (AAT) was introduced at Picatinny Arsenal in 1914. Under AAT, master samples taken from gun propellant lots are sequentially stored in heating chambers at 65.5 ^{0}C to measure the red nitrogen oxide fume time. Once the fume time is observed, it is related to the age of propellant stockpiles.

The current methods to estimate the shelf-life of a group of similar gun propellants are based on a fixed effects linear regression model for the fume time. The shortcomings of the current estimation methods are such that the relationship between the fume time and propellant age may not necessarily be a linear one and the fixed effects model does not take into account the potential lot-to-lot variation. To consider potential lot-to-lot variation, a random effects regression model (RERM) is required.

To understand the state-of-art estimation of the safe shelf-life of gun propellant, four technical reports are briefly reviewed. Furthermore, a preliminary analysis is conducted to understand the basic characteristics of the master samples and fleet return data sets of 5"/54 NACO gun propellant provided by Naval Surface Warfare Center (NSWC). By analyzing the fitted models and validating the model

assumptions, the standard linear regression model based on the reduced data set is selected to further investigate a random effects regression model analysis.

To consider a potential lot-to-lot variation when estimating the shelf-life, first the RERM is developed using a two-stage analysis. Next, a combined mixed effects model is estimated by employing both the maximum likelihood (ML) and restricted maximum likelihood (REML) methods. These estimators are used to define the shelf-life of the group of similar lots. The estimated model is used to predict not only the shelf-life of a group of the similar lots but also that for each individual lot by a shrinkage procedure.

The results indicate that, first, the claimed shelf-life (35 years) for the NACO propellant lots is not adequate because it overestimates the shelf-life for NACO propellant lots by almost 10 years. Therefore, it requires amendment. Next, the group shelf-life estimated can be relatively conservative compared to the individual shelf-lives. In view of potential opportunity loss due to individual lots discarded when they are still safe, use of individual shelf-life is recommended over the group shelf-life when the management of individual lots is expected to cost less than that of bulk management.

There is a nonlinear decreasing trend in fume time in some individual lots as the propellant ages. This raises questions about the approaches that use traditional linear least squares analysis of fume time data to predict safe shelf-life. This thesis recommends that a nonlinear regression analysis approach be conducted for some individual propellant lots.

I. INTRODUCTION

A. BACKGROUND

Most gun ammunition is stored at depots for a long time before being used. In order to avoid potential disaster due to storing unstable gun propellant, the shelf-life of a gun propellant stockpile should be accurately estimated. Typically, the shelf-life of gun propellant stockpile is defined as the age at which 5% of the gun propellant is unstable. The estimated shelf-life can also be used to efficiently minimize the ammunition management costs for distribution, maintenance, consumption and disposal.

In the early 1900's, real time storage inspection was used to estimate the safe shelf-life of gun propellant lots produced in the U.S.A. However, under the real time storage inspection, it took a long time to find visible changes in stability of propellent. In an effort to reduce the length of time required for the real time storage inspection, an Accelerated Aging Test (AAT) was introduced at Picatinny Arsenal in 1914 [Ref.3]. For AAT, master samples are collected for testing purposes from each gun propellant lot produced in the U.S.A. over the past sixty years. Each master sample consists of 5 pounds of propellant from each lot.

Under the AAT, forty-five grams of propellant are taken from each master sample and are stored in heating chambers at 65.5 °C until red nitrogen oxide fumes appear. The time it takes to observe such oxide fumes is called the fume time. This fume time is recorded with the corresponding propellant age. If the fume time falls below thirty days, the propellant is considered unsafe and the lot is recommended to be condemned. A fresh batch of propellant is then taken from ambient storage and the AAT is repeated. In general, it is assumed that fume time decreases as propellants age. In order to validate the results obtained from the master samples, the

AAT is employed on the fleet-return data of gun propellant lots stored in the active depots.

The classical methods used to estimate the shelf-life of a group of similar gun propellants is based on a fixed effects linear regression model. The fume time data are aggregated over a group of similar lots and a linear model for fume time is fitted against propellant age. The safe shelf-life of current methods is then estimated as the time period at which a 95% one sided lower prediction limit for the fume time curve intersects the acceptable lower specification level of thirty days of fume time. The shortcomings of the current estimation method are such that the relationship between the fume time and propellant age may not necessarily be a linear one and the fixed effects model does not take into account the potential lot-to-lot variation. Other relationships in addition to the linear model should be examined. In order to consider potential lot-to-lot variations, a random effects model is required.

To understand the state-of-art related to the estimation of the safe shelf-life of gun propellant, four technical reports are briefly reviewed.

B. LITERATURE REVIEW

In the report entitled "Prediction of Safe Life of Propellants" [Ref. 1], the nature of 65.5°C surveillance test and the propellant chemical deterioration are briefly discussed. In this study, it is recognized that the measurement of residual stabilizer content offers the best means of establishing the stability potential of propellant. This report also discusses the results obtained from the standard artillery propellant when exposed to the aging test at various temperatures.

The measurements of residual stabilizer content versus time were used as the proxy for propellant deterioration. For

interpretation of this deterioration phenomena, Berthelot's Law was employed. It demonstrated that a family of straight lines can be plotted characterizing the length of time necessary at the various test temperatures to obtain a given variation of stabilizer content. Finally, by establishing realistic cut-off points regarding stabilizer content in a given storage temperature, propellant safe life was estimated.

Secondly, the report entitled "Type Life Program History, Philosophy, Accomplishment, And Future" [Ref. 2] introduces the concept of the type life test. The type life test is derived from the AAT approach and takes into account a more realistic temperature. In this study, accelerated aging techniques are used to observe the aging mechanisms and characteristics of propulsion ordnance along with associated components. They are also use to determine how age affects the performance of the units.

The type life temperature-time profile defined in this study is based on the "hot-month" concept, and differs from the AAT and its use of constant testing temperature. The concept is briefly stated as follows:(1)propellants are a chemical system;(2)chemical systems deteriorate more rapidly at elevated temperatures; and (3)in a normal year's time, the deterioration occurs only during the summer months.

Based on "hot-month" concept, the engineers establish a temperature-time profile termed the "compressed-ambient cycle" and consisting of 26 weeks. This profile was designed to simulate the four seasons of the year and is related to the aging that normally occurs during magazine storage in the U.S.A. There is a 16-week period at 100 °F (38°C) representing a long severe summer time, two 3-week periods at 70 °F representing spring and autumn, and a 4-week period at 40 °F representing winter. The compressed-ambient cycle (26 weeks or 1/2 year) simulates one year of magazine storage; therefore there is a 2:1 (i.e.52 weeks:26 weeks) aging ratio. The main

purpose of this report was to evaluate the effect of the loss of nitroglycerin from the propellant into the inhibitor. This loss results in propellant cracking and/or a reduction in propulsion performance due to a loss of energy.

Thirdly, a report entitled "Statistical Analysis of ARDEC'S Fume Data Base" [Ref. 3] describes the procedure for AAT based on the master sample surveillance program in detail. Three types of propellants were used separately based on single and double base propellants. The single and double bases are terms of ammunition, distinguished by the methods of ignition. The single base propellant can be ignited by either mechanical or electrical means. The double base propellant can be ignited using both methods. In this study, fume times are plotted against sampling times for each propellant form. Not only is the first order linear relationship examined with this approach but also other relationships such as polynomial regressions, logarithmic and exponential transformation are considered to establish fume time behavior over the life of the propellant.

Finally, a study entitled "Long-Term Stability of Navy Gun Propellants" [Ref. 4] was initiated to standardize the requirements of safety surveillance testing between the Army and Navy. This was necessary because current Navy surveillance testing includes a 65.5 °C oven fume test (AAT) on single-base propellants and double-base propellants with less than or equal to 10% nitroglycerin content. Additionally, propellants must take more than 30 days to fume to be considered stable. In contrast, the Army's disposition criteria is based on a stabilizer analysis of less than 0.2% nitroglycerin content. Because a substantial quantity of Navy propellant is being currently stored in Army depots, non-standardization complications commonly occur. This study was tasked by Naval Sea Systems Command(NSSC) to determine the critical level of propellant stabilizer for selected

propellants of interest to the Navy and to the Marine Corps, to develop the best test methods to evaluate that level of criticality for Navy propellants, and to apply the data for standardization.

The test was conducted on propellants removed from loaded ammunition. Each sample was subjected to chemical, kinetic, and physical properties analyses. As a result of these analyses, it was recommended to continue use of the 65.5 °C oven fume test because it is the procedure that provides the most complete coverage of propellant in storage worldwide. The Navy has a large historical database of fume data, and statistical analysis of the data provides an inexpensive, comprehensive means of insuring that Navy-developed propellant is safe for worldwide storage.

In summary, the master sample surveillance test is recommended for continuous use to determine safety of the gun propellant in storage. It is proposed that the relationship between the fume time and propellant age may not be limited to a linear relationship. The management of data structure and the data transformation (e.g. logarithmic or reciprocal) is required. Additionally, no potential lot-to-lot variation in shelf-life estimation was examined in the four technical reports summarized here.

C. THE SCOPE OF THESIS

In this thesis, the master sample surveillance test data set is first examined using several fixed effects regression models in an attempt to find the relationship between the fume time and propellant age. Then, a random effects regression model is introduced to take into account a lot-to-lot variation in fitting the relationship between the fume time and propellant age. Finally, results are discussed and recommendations are made.

II. FIXED EFFECTS REGRESSION MODEL

A. DATA DESCRIPTION

A preliminary analysis is conducted to understand the basic characteristics of the master sample data set provided by Naval Surface Warfare Center (NSWC). The data set was collected based on the oven test data consisting of 28 lots of master samples of 5"/54 NACO gun propellant. They were manufactured at Badger Army Ammunition Plant (BAAP), Baraboo, Wisconsin between 1972 and 1974. The NACO is a generic term of NAvy COol representing a family of cool-burning, single-base propellants. These propellants are made from low-nitration nitrocellulose with a coolant, a stabilizer, a decoppering agent, and a flash suppressant added.

First by subtracting the manufacturing date (see Appendix A) from the date when a 45g propellant batch was sent to the heating chamber, the age is computed for each propellant lot. The data of the fume time and propellant age is shown in computer program list in Appendix B. Both descriptive statistics of fume time and propellant age are displayed in Table 1. In summary, 221 observations of fume time and propellant age had been taken from 28 propellant lots. The maximum (1492 days) and minimum (19 days) of fume time are observed during 6813 days of propellant age. Next, in an attempt to find the relationship between the fume time and propellant age, the fume time is plotted against the propellant age in Figure C.1. It is observed that there is an unexpected increasing trend in fume time over propellant age up to 1100 days. This unexpected pattern may be due to the type of ammunition, shape of propellant container or caused by some unknown chemical reaction. [Ref. 3]

Descriptive Statistics For Raw Data		
manufacture date		min 1972 max 1974
number of lot		28
number of repeated measurements		221
fume time in days	mean	802.61
	std	222.159
	max	1492
	min	19
propellant age in days	mean	3488.824
	std	2020.824
	max	6813
	min	0

Table 1. Descriptive Statistics For Raw Data

B. STANDARD LINEAR REGRESSION MODEL I

Regardless of such unexpected pattern, according to the current practice, standard linear regression model I, is applied to fit the fume time (Y) against propellant age (X):

$$Y = B_0 + B_1 X + \epsilon \tag{1}$$

where B_0 and B_1 represent the expected initial condition and deterioration rate of stability of gun propellant, and ϵ is the random error which follows a Normal $(0,\tau^2)$ distribution.

The predicted value ($\tilde{Y}_*$) at $X=X_*$ is

$$\tilde{Y}_* = \hat{B}_0 + \hat{B}_1 X_* \tag{2}$$

where $\hat{B}_0$ and $\hat{B}_1$ are the ordinary least square estimators of B_0 and B_1. Subsequently a 90% prediction interval for a fume time at given propellant age (X_*) is

$$\tilde{Y}_* \pm t\ (\ 0.95\ ,\ N-2\ )\ SEPRED\ (\ \tilde{Y}_*\ |\ X_*\ ), \tag{3}$$

where N is the total number of observations, t(0.95,N-2) is the 95th percentile of the t distribution with N-2 degrees of freedom. $SEPRED\ (\tilde{Y}_*\ |X_*)$ is the standard error of predicted $\hat{Y}_*$ at $X=X_*$:

$$SEPRED\ (\ \tilde{Y}_*\ |\ X_*\ ) = \hat{\tau}\ [\ 1 + \frac{1}{N} + \frac{(\ X_* - \overline{X}\ )^2}{\sum (\ X_* - \overline{X}\ )^2}\ ]^{\frac{1}{2}}. \tag{4}$$

Based on this information, the group shelf-life (t_{sl}) can be estimated by checking the time period when the 95% one-sided lower prediction limit intersects 30 days of fume time:

$$30 = \hat{B}_0 + \hat{B}_1\ t_{sl} - t\ (\ 0.95\ ,\ N-2\ )\ SEPRED\ (\ \tilde{Y}_*\ |\ t_{sl}\ ). \tag{5}$$

Next, actual data obtained from the NACO propellant surveillance test is used to fit the model and to estimate the shelf-life. The fitted model based on (2) is given in Table

Standard Linear Regression Model I	
$\hat{B}_0$	1045.510
$\hat{B}_1$	-0.070
$SE(\hat{B}_0)$	23.171
$SE(\hat{B}_1)$	0.006
R-square	0.4009
lack of fit test	degrees of freedom: 200,19; P value = 0
Normal distribution test (Kolmogorov-Smirnov Test)	P value = 0.004 **not Normal**
estimated shelf-life	10400 days (28.49 years)

Table 2. Fitted Standard Linear Regression Model I

2 and displayed as a solid line in Figure C.1. A 90% two-sided prediction interval (solid curves) is overlaid in Figure C.1. The resultant shelf-life based on (5) turns out to be 10400 days (28.49 years) as shown in Figure C.2.

The residual analysis $(r_i = Y_i - \hat{Y}_i \text{ , for } i = 1 \ldots N)$ for standard linear regression model I is shown in Figure C.3. Residual pattern indicates nonlinearity and nonconstant variance. Also, the Kolmogorov-Smirnov test statistic shown in Table 2 indicates that the residuals do not follow a normal distribution.

For further data analysis, the data set that covers from zero to 1100 days of propellant age is deleted, because the pattern is unstable during this early stage and this pattern

is not expected to last longer than 1100 days. Furthermore, after 1100 days of propellant age, the fume time tends to decrease as propellants age and this pattern is more consistent. Therefore, the use of the remaining data set appears to be reasonable for the shelf-life estimation. The following three regression models are employed (1) standard linear regression model II (delete observations that correspond to propellant age below 1100 days and use standard linear regression model I);(2) linearizing transformation model I (transform propellant age to 1/ propellant age); and (3) linearizing transformation model II (transform fume time to log(fume time)). Graphical method precedes statistical analysis.

C. STANDARD LINEAR REGRESSION MODEL II

The standard linear regression model II is essentially the same as the standard linear regression model I(1). However, parameters are estimated based on a reduced data set. After deleting the early age, the fume time is plotted against propellant age in Figure C.4. The results of model fit are shown in Table 3. The predictive value (middle solid line) and a 90% two-sided prediction interval for a fume time (solid curves) are overlaid in Figure C.4.

The residual analysis for the standard linear regression model II is shown in Figure C.5. In general, the plot of residuals against fitted values over fume time between zero day and 750 days (propellant age above 4500 days) does not show any systematic features and they appear to have a common variance. But, a minor nonrandom pattern is observed when the fume time exceeds 750 days (propellant age below 4500 days). Also, based on the p-value associated with Kolmogorov-Smirnov test shown in Table 3, we can conclude the residual of this model follows a normal distribution.

Standard Linear Regression Model II	
$\hat{B}_0$	1388.452
$\hat{B}_1$	-0.141
$SE(\hat{B}_0)$	20.612
$SE(\hat{B}_1)$	0.005
R-square	0.838
lack of fit test	degrees of freedom: 167,11; P value = 0.001
Normal distribution test (Kolmogorov-Smirnov Test)	P value = 0.458 **Normal**
estimated shelf-life	8469 days (23.20 years)

Table 3. Fitted Standard Linear Regression Model II

By checking the level of 95% one-sided lower prediction limit for a fume time that intersects 30 days of fume time in Figure C.6, the estimated group shelf-life turns out to be 8469 days (23.20 years).

D. LINEARIZING TRANSFORMATION MODEL I

In linearizing transformation model I, it is assumed that the fume time is linearly related to reciprocal of propellant age:

$$Y = B_0 + B_1 \left(\frac{1}{X} \right) + \epsilon \qquad (6)$$

The fume time is plotted against propellant age in Figure C.7.

The results of a model fit are shown in Table 4. The predicted value for the fume time $\tilde{Y}_* = \hat{B}_0 + \hat{B}_1 \left(\frac{1}{X_*} \right)$ is shown

Linearizing Transformation model I	
$\hat{B}_0$	411.738
$\hat{B}_1$	1315827.380
$SE(\hat{B}_0)$	17.420
$SE(\hat{B}_1)$	51308.706
R-square	0.787
lack of fit test	degrees of freedom: 167,11; P value = 0
Normal distribution test (Kolmogorov-Smirnov Test)	P value = 0.02948 **not Normal**
estimated shelf-life	∞

Table 4. Fitted Linearizing Transformation Model I

as a middle curve and a 90% two-sided prediction interval for a fume time are overlaid in Figure C.7.

It is interesting to note that the 95% one-sided lower prediction limit for predictive fume time converges to around 400 days as the propellant age continuously increases. Therefore, the safe shelf-life of gun propellant based on linearizing transformation model I cannot be estimated or can be considered as infinity.

The residual analysis for the linearizing transformation model I is shown in Figure C.8. In general, the plot of

residuals against fitted values over fume time between 400 days and 800 days (propellant age above 4000 days) does not show any systematic features and they appear to have a common variance. But a nonrandom pattern is observed when the fume time exceeds 800 days (propellant age below 4000 days). This phenomenon is similar to the standard linear regression model II (see Figure C.5). Also, based on the p-value associated with Kolmogorov-Smirnov test shown in Table 4, we can conclude that the residual of this model does not follow a normal distribution.

E. LINEARIZING TRANSFORMATION MODEL II

In linearizing transformation model II, it is assumed that the propellant age is linearly related to log fume time:

$$LOG\ (Y) = B_0 + B_1\ X + \epsilon. \tag{7}$$

The fume time is plotted against propellant age in Figure C.9. The results of a model fit are shown in Table 5. The predicted value for the fume time $\tilde{Y}_* = EXP\ (\hat{B}_0 + \hat{B}_1 X_*)$ and a 90% two-sided prediction interval for a fume time are overlaid in Figure C.9.

The residual analysis for the linearizing transformation model II is shown in Figure C.10. In general, the plot of residuals against fitted values over fume time between zero day and 6.6 days (propellant age above 4000 days) does not show any systematic features and they appear to have a common variance. But a nonrandom pattern is observed when the fume time exceeds 6.6 days (propellant age below 4000 days). This phenomenon is also similar to the previous two models (see Figure C.5 and Figure C.8). Also, based on the p-value associated with Kolmogorov-Smirnov test shown in Table 5, we

Linearizing Transformation model II	
$\hat{B}_0$	7.38
$\hat{B}_1$	-1.79 E-4
$SE(\hat{B}_0)$	5.75 E-2
$SE(\hat{B}_1)$	1.29 E-5
R-square	0.520
lack of fit test	degrees of freedom: 167,11; P value = 0
Normal distribution test (Kolmogorov-Smirnov Test)	P value = 1.0E-11 **not Normal**
estimated shelf-life	17400 days (47.67 years)

Table 5. Fitted Linearizing Transformation Model II

can conclude that the residual of this model does not follow a normal distribution.

By checking the level of the 95% one-sided lower prediction limit of a fume time that intersects 30 days of fume time in Figure C.11, the estimated group shelf-life turns out to be 17400 days (47.67 years).

In summary, by analyzing the fitted models and validating the model assumptions of the various fixed effects regression models, the effective distribution data model appears to be the standard linear regression model II based on the reduced data. This model is compared to the model fit based on the fleet return data.

F. STANDARD REGRESSION MODEL BASED ON FLEET RETURN DATA

This model is based on the oven test data consisting of 28 lots of fleet return data of 5"/54 NACO gun propellant. The descriptive statistics of fume time and propellant age are displayed in Table 6. In summary, 204 observations of fume

Descriptive Statistics		
manufacture date		min 1972 max 1974
number of lot		28
number of repeated measurements		202
fume time in days	mean	702.12
	std	85.46
	max	993
	min	479
propellant age in days	mean	2822
	std	1388.8
	max	5074.5
	min	361

Table 6. Descriptive Statistics For Fleet Return Data

time and propellant age had been taken from 28 propellant lots. The maximum (993 days) and minimum (479 days) of fume time are observed during 5074.5 days of propellant age. The fume time is plotted against propellant age in Figure C.12. The results of model fit which is based on standard linear

Standard Linear Regression Model	
$\hat{B}_0$	823.375
$\hat{B}_1$	-0.042
$SE(\hat{B}_0)$	9.793
$SE(\hat{B}_1)$	0.003
R-square	0.422
lack of fit test	degrees of freedom: 127,73; P value = 0.0000
Normal distribution test (Kolmogorov-Smirnov)	P value = 0.0040 **not Normal**
estimated shelf-life	15700 days (43 years)

Table 7. Fitted standard Linear Regression Model Based on Fleet Return Data

regression model I are shown in Table 7. The predicted value (middle solid line) and a 90% prediction interval for a fume time (solid curves) are overlaid in Figure C.12. The resultant shelf-life based on (5) of this model turns out to be 15700 days (43 years) as shown in Figure C.13.

The residual analysis for this model is shown in Figure C.14. There is a right-opening megaphone pattern in Figure C.14. This residual pattern observed shows nonconstant variance. Also, the Kolmogorov-Smirnov test statistic shown in Table 7, indicates that the residuals of this model do not follow a normal distribution. Two remedies for nonconstant variance (Weisberg [Ref. 8]) are made using weighted least square estimation and power transformation:

1. Weighted Least Square Model

The first remedy is the weighted least square model. The variance of the random error in the standard linear regression model I (1) appears to increase as X increases, that is, τ=KX where K is a positive constant. Therefore, the model (1) is modified as follows:

$$\frac{Y}{X} = \frac{B_0}{X} + B_1 + \frac{\epsilon}{X}. \qquad \textbf{(8)}$$

The residual analysis for this model is shown in Figure C.15. It still shows a right-opening megaphone pattern in Figure C.15 and the suggested weighted least square method is not adequate.

2. Power Transformation Model

The second remedy is to transform the response Y via a variance stabilizing transformation. By checking the residual pattern of several transformation models, the following transformation model appears to satisfy the assumption of common variance (see Figure C.16):

$$Y^{-3} = B_0 + B_1 X + \epsilon. \qquad \textbf{(9)}$$

The fume time is plotted against propellant age in Figure C.17. The results of a model fit are shown in Table 8. The prediction fume time $\tilde{Y} = (\hat{B}_0 + \hat{B}_1 X_*)^{-\frac{1}{3}}$ and a 90% prediction interval for a fume time are overlaid in Figure C.17.

The p-value associated with Kolmogorov-Smirnov test of residual of model (9) indicates that the residual of this model follows a normal distribution.

Power Transformation model	
$\hat{B}_0$	1.75 E-9
$\hat{B}_1$	4.87 E-13
$SE(\hat{B}_0)$	1.26 E-10
$SE(\hat{B}_1)$	4 E-14
R-square	0.425
lack of fit test	degrees of freedom: 127,73; P value = 0.001
Normal distribution test (Kolmogorov-Smirnov)	P value = 0.18964 **Normal**
estimated shelf-life	∞

Table 8. Fitted Power Transformation Model

The shelf-life is estimated based on the following formula:

$$30 = [\hat{B}_0 + \hat{B}_1\, t_{s1} - t(\,0.95\,,\,N\text{-}2\,)\ SEPRED(\,\tilde{Y}_* \mid t_{s1}\,)\,]^{-\frac{1}{3}}. \quad \textbf{(10)}$$

The estimated group shelf-life turns out to be infinite.

This shelf-life estimation based on fixed effects regression models does not take into account the lot-to-lot variation. Apparently potential lot-to-lot variation is supported by individually fitted models based on the Standard Linear Regression Model I, II and fleet return data as shown

in Figures C.18, C.19, C.20. Additionally, this variation can be seen by checking linearizing transformation model I after smoothing the data following Lowess procedure [Ref. 9] as shown in Figure C.21.

In the next chapter, standard linear regression model II will be further investigated for a random effects regression model analysis.

III. RANDOM EFFECTS REGRESSION MODEL

A. INTRODUCTION

The current methods used to estimate the shelf-life of gun propellant is based on the standard linear regression model I. In this fixed effects linear regression model, the expected initial fume time (B_0) and deterioration rate (B_1) are assumed to be constant for all gun propellant lots. However, in practice, the performance varies among lots even though a group of lots is manufactured based on the same technique. This kind of phenomenon can be observed in the individually fitted models in Figure C.18 to C.21. The lot-to-lot variation is observed not only in the intercepts (expected initial condition) but also in slopes (deterioration rate) among a group of similar lots. The current methods does not take into account this kind of constant. In order to consider potential lot-to-lot variation within the group, a random effects (RERM) (or mixed effects) regression model is necessary.

In this chapter, first, the RERM will be introduced based on a two-stage analysis. Next, a combined two-stage model (mixed effects model) will be fitted based on the reduced data of the master samples provided by NSWC. In order to obtain the estimators of unknown parameters in mixed effects model, the maximum likelihood estimation (MLE) and restricted maximum likelihood (REML) will be employed. These estimators are, then, used to defined the shelf-life of the group of similar lots that takes into account the lot-to-lot variation. Furthermore, a shrinkage procedure will also be introduced to estimate the shelf-life of an individual lot.

B. MODEL

1. Within-Lot Model

In order to estimate the deteriorating patterns of gun propellant lots over storage time, each 45g batch is taken from 5 pound master sample of lot i and its ageing is accelerated at the heating chamber at 65.5 ^{0}C. The fume time (y_{ij}) of lot i (i=1...N) is recorded with the corresponding propellant age (t_{ij}) (j=1...n_i) whenever the red nitrogen oxide fume appears. Once repeated measurements are obtained, we attempt to find the relationship between the fume time and propellant age. The following within-lot model is used to describe the relationship between the fume time and the age of a lot:

For i=1...N and j=1...n_i

$$y_{ij} = \alpha_{0i} + \alpha_{1i}\, t_{ij} + \epsilon_{ij} \tag{11}$$

where α_{0i} and α_{1i} represents the expected initial condition and deterioration rate of the stability of propellant lot i, and ϵ_{ij} is the random error which is assumed to follow a independent N(0, τ^2).

2. Between-Lot Model

In order to take into account the lot-to-lot variation among intercepts (expected initial condition) and slopes (deterioration rate) of a group of lots, we assume the following between-lot model:

$$\begin{aligned} \alpha_{0i} &= w_i\, \gamma_0 + \delta_{0i} \\ \alpha_{1i} &= w_i\, \gamma_1 + \delta_{1i} \end{aligned} \tag{12}$$

where w_i is a $1 \times k$ vector representing values of K factors that may cause varying α_{0i} and α_{1i} . The γ_0 and γ_1 are $k \times 1$ vectors of regression coefficients for α_{0i} and α_{1i}, respectively. The δ_{0i} and δ_{1i} are assumed to follow independent $N(0,\Sigma)$, where $\Sigma = \begin{bmatrix} \sigma_0^2 & \sigma_{01}^2 \\ \sigma_{01}^2 & \sigma_1^2 \end{bmatrix}$. Note that for AAT, the master samples are put in the constant temperature condition (65.5 ^{0}C) and w_i in this case is 1.

When α_{0i} and α_{1i} in (11) are substituted with those in (12), a combined mixed effects model and is formulated as follows:

For $i=1...N$ and $j=1...n_i$,

$$
\begin{aligned}
y_{ij} &= (w_i \gamma_0 + \delta_{0i}) + (w_i \gamma_1 + \delta_{1i}) \ t_{ij} + \epsilon_{ij} \\
&= (w_i \gamma_0 + w_i \gamma_1 t_{ij}) + (\delta_{0i} + \delta_{1i} t_{ij}) + \epsilon_{ij} \qquad \textbf{(13)} \\
&= (w_i , w_i t_{ij}) \begin{pmatrix} \gamma_o \\ \gamma_1 \end{pmatrix} + (1 , t_{ij}) \begin{pmatrix} \delta_{oi} \\ \delta_{1i} \end{pmatrix} + \epsilon_{ij} .
\end{aligned}
$$

This mixed effects model can be re-written using matrix notation:

$$
y = X\beta + Z\nu + e , \qquad \textbf{(14)}
$$

where y is a $n_i \times 1$ vector of y_{ij}; β is a $2K \times 1$ coefficient vector$(\gamma_0 , \gamma_1)'$ associated with a $\sum_{i=1}^{N} n_i \times 2K$ matrix of X consisting of

$(w_i\ ,\ w_i t_{ij})$; υ is a $2n_i \times 1$ vector of $(\delta_{0i}\ ,\ \delta_{1i})'$ associated with a known $\sum_{i=1}^{N} n_i \times 2n_i$ block diagonal matrix Z of $(1\ ,\ t_{ij})$, and e is an $\sum_{i=1}^{N} n_i \times 1$ vector of random error. Assume that υ and e are uncorrelated and have expections 0 and variances G and R, respectively, where both G and R are nonsingular. applying these assumptions to (14) leads to

$$E(y) = X\beta$$
$$Var(y) = V = ZGZ' + R\ , \tag{15}$$

where G is a $2n_i \times 2n_i$ block diagonal matrix consisting of $\begin{bmatrix} \sigma_0^2 & \sigma_{01}^2 \\ \sigma_{01}^2 & \sigma_1^2 \end{bmatrix}$ and $R = Var(e) = \tau^2 I_{\sum_{i=1}^{N} n_i}$.

When G and R (and hence V) are known, estimates of both β and the realized value of υ are

$$\hat{\beta} = [\hat{\gamma}_0\ ,\ \hat{\gamma}_1]' = (X'V^{-1}X)^{-1}X'V^{-1}y$$
$$\hat{\upsilon} = [\hat{\upsilon}_1, \ldots \hat{\upsilon}_N]' = [\hat{\delta}_{01}, \hat{\delta}_{11}, \ldots \hat{\delta}_{0N}, \hat{\delta}_{1N}]' = GZ'V^{-1}(y - X\hat{\beta}) \tag{16}$$

respectively, where $\hat{\beta}$ is the best linear unbiased estimator (BLUE) of β, and $\hat{\upsilon}$ is the best linear unbiased predictor (BLUP) of υ. They can also be obtained from Henderson's (1984) mixed model equation [Ref. 10]:

$$\begin{bmatrix} X'R^{-1}X & X'R^{-1}Z \\ Z'R^{-1}X & Z'R^{-1}Z+G^{-1} \end{bmatrix} \begin{bmatrix} \beta \\ \hat{v} \end{bmatrix} = \begin{bmatrix} X'R^{-1}y \\ Z'R^{-1}y \end{bmatrix} \tag{17}$$

where $\beta = [\hat{\gamma}_0 \ , \ \hat{\gamma}_1]'$ and $\hat{v} = [\hat{\delta}_{01} \ , \ \hat{\delta}_{11}, \ldots \hat{\delta}_{0N} \ , \ \hat{\delta}_{1N}]'$. When G and R (and hence V) are unknown, the estimator of V can be obtained by maximizing the logarithm of likelihood function [Ref. 10]

$$\log L = -\frac{1}{2} N \log 2\pi - \frac{1}{2} \log|v| - \frac{1}{2} (y - X\beta)' V^{-1} (y - x\beta) . \tag{18}$$

The ML estimators $\hat{V}$ and β can be obtained by solving the following equations simultaneously

$$X' \hat{V}^{-1} X \beta = X' \hat{V}^{-1} y \tag{19}$$

and

$$tr(\hat{V}^{-1} Z_i Z_i') = (y - X\beta)' \hat{V}^{-1} Z_i Z_i' \hat{V}^{-1} (y - X\beta) \qquad for \ i = 0, 1, \ldots N, \tag{20}$$

where Z_i is a $\sum_{i=1}^{N} n_i \times 2n_i$ block diagonal matrix of $(1, t_{ij})$ and

$tr(\hat{V}^{-1} Z_i Z_i')$ is trace of the $\sum_{i=1}^{N} n_i \times \sum_{i=1}^{N} n_i$ matrix $(\hat{V}^{-1} Z_i Z_i')$ that

denotes the sum of the diagonal entries. An algebraically simpler expression for (20) is derived by defining

$$P = V^{-1} - V^{-1}X(X'V^{-1}X)^{-1}X'V^{-1}. \tag{21}$$

Then from (19) it is clear that for $\hat{P}$ being P with V replaced by $\hat{V}$

$$\hat{V}^{-1}(y - X\boldsymbol{\beta}) = \hat{P}y, \tag{22}$$

so that the ML estimators of $\hat{V}$ and $\boldsymbol{\beta}$ also can be re-written by solving the following ML equations simultaneously

$$X' \hat{V}^{-1} X \boldsymbol{\beta} = X' \hat{V}^{-1} y \tag{23}$$

and

$$\{_c\ tr(\hat{V}^{-1}Z_iZ_i')\ \}_{i=0}^{N} = \{_c\ y'\hat{P}Z_iZ_i'\hat{P}y\ \}_{i=0}^{N}\ , \tag{24}$$

where $\{_c\ tr(\hat{V}^{-1}Z_iZ_i')\ \}_{i=0}^{N}$ is (N+1)×1 column vector with elements which are the trace of the matrix $(\hat{V}^{-1}Z_iZ_i')$, for i=0,1,...N, respectively.

Also, the estimator of V can be obtained by restricted maximum likelihood (REML). A basic idea of REML estimation is that of estimating variance components based on residuals calculated after fitting by ordinary least squares just the fixed effects part of the model. The REML estimator can also by viewed as maximizing a marginal likelihood. The REML equations can therefore be derived from the ML equations of (24). By making suitable replacements, the REML estimator $\hat{V}$

can be obtained by solving the following equation:

$$\{ tr[(K'\hat{V}K)^{-1}K'ZZ'K]\}_{i=0}^{r} = \{ y'K(K'\hat{V}K)^{-1}K'ZZ'K(K'\hat{V}K)^{-1}K'y \}_{i=0}^{r} \tag{25}$$

with $P = K(K'VK)^{-1}K'$ where $K'X=0$.

When the resultant $\hat{V}$ is found by solving the ML equations ((19),(20)) or REML equation (25), the Henderson's mixed model equation can be re-written as follows:

$$\begin{bmatrix} X'\hat{R}^{-1}X & X'\hat{R}^{-1}Z \\ Z'\hat{R}^{-1}X & Z'\hat{R}^{-1}Z+\hat{G}^{-1} \end{bmatrix} \begin{bmatrix} \hat{\beta} \\ \hat{v} \end{bmatrix} = \begin{bmatrix} X'\hat{R}^{-1}y \\ Z'\hat{R}^{-1}y \end{bmatrix} . \tag{26}$$

Finally, $\hat{\beta} = [\hat{\gamma}_0 , \hat{\gamma}_1]'$ and $\hat{v} = [\hat{\delta}_{01}, \hat{\delta}_{11}, \ldots \hat{\delta}_{0N}, \hat{\delta}_{1N}]'$ substitutes $\beta = [\gamma_0, \gamma_1]'$ and $v = [\delta_{01}, \delta_{11}, \ldots \delta_{0N}, \delta_{1N}]'$ in (14) as follows:

$$\hat{y} = X \hat{\beta} + Z \hat{v} . \tag{27}$$

In fume time analysis, the test temperature condition is constant and W_i in this analysis is 1. Therefore, $X_{ij}=(W_i, W_i t_{ij})=(1,t_{ij})$ and $Z_{ij}=(1,t_{ij})$. Then (27) becomes

$$
\begin{aligned}
\hat{y} &= X\hat{\beta} + Z\hat{\nu} = Z\hat{\beta} + Z\hat{\nu} \\
&= Z\hat{\beta} + Z(\hat{G}Z'\hat{V}^{-1}(y - Z\hat{\beta})) \\
&= Z\hat{\beta} + Z\hat{G}Z'\hat{V}^{-1}y - Z\hat{G}Z'\hat{V}^{-1}Z\hat{\beta} \qquad (28) \\
&= (I - Z\hat{G}Z'\hat{V}^{-1})Z\hat{\beta} + (Z\hat{G}Z'\hat{V}^{-1})y \\
&= (I - H)\hat{E}(y) + Hy
\end{aligned}
$$

where $H=Z\hat{G}Z'\hat{V}^{-1}$ is the weighted of mean of $\hat{E}[y]$ and y. The resulting $\hat{y}$ is called a shrinkage estimator.

The predicted value of fume time $(\tilde{y}_{ij})$ of lot i at t_{ij} can be estimated as

$$
\tilde{y}_{ij} = x_{ij}\begin{pmatrix}\hat{\gamma}_0\\ \hat{\gamma}_1\end{pmatrix} + z_{ij}\begin{pmatrix}\hat{\delta}_{0i}\\ \hat{\delta}_{1i}\end{pmatrix} = (1, t_{ij})\begin{pmatrix}\hat{\gamma}_0\\ \hat{\gamma}_1\end{pmatrix} + (1, t_{ij})\begin{pmatrix}\hat{\delta}_{0i}\\ \hat{\delta}_{1i}\end{pmatrix}. \qquad (29)
$$

A 90% prediction interval for a fume time at given propellant age t_{ij} is

$$
\tilde{y}_{ij} \pm t\,(0.95, N-2)\; SEPRED\,(\tilde{y}_{ij} \mid t_{ij}) \qquad (30)
$$

where

$$
SEPRED(\tilde{y}_{ij}|t_{ij}) = \left[(1, t_{ij})\hat{V}(\hat{\beta}_i)(1, t_{ij})' + (1, t_{ij})\hat{V}(\hat{\nu}_i)(1, t_{ij})' + \hat{\tau}^2\right]^{\frac{1}{2}}; \qquad (31)
$$

and $\hat{V}(\hat{\beta}_i)$ and $\hat{V}(\hat{\nu}_i)$ is the i^{th} block diagonal matrices of

$$\hat{V}(\hat{\beta}) = (X' \hat{V}^{-1} X)^{-1};$$

$$\hat{V}(\hat{v}) = \hat{G} Z' \hat{V}^{-1} \hat{G} Z' . \tag{32}$$

In order to estimate deteriorating patterns of a group of similar lots, one would be interested in the shelf-life of the group of similar lots (t_{sl}) that takes into account the lot-to-lot variation. It can be obtained as follows by only considering the group effects:

$$30 = x_{ij} \hat{\beta} - t\ (0.95\ , N\text{-}2\ )\ SEPRED_{(group)}\ (\tilde{y} \mid t_{sl})$$

$$= (1, t_{sl}) \begin{pmatrix} \hat{\gamma}_0 \\ \hat{\gamma}_1 \end{pmatrix} - t\ (0.95, N\text{-}2)\ SEPRED_{(group)}\ (\tilde{y} \mid t_{sl}) \tag{33}$$

where $SEPRED_{(\mathrm{group})}\ (\tilde{y} \mid t_{sl})$ is the standard error of predicted $\hat{y}$ of a group given by

$$SEPRED_{(\mathrm{group})}\ (\tilde{y} | t_{ij}) = \left[\ (1, t_{ij}) \hat{V}(\hat{\beta}_i) (1, t_{ij})' + \hat{\tau}^2\ \right]^{\frac{1}{2}} . \tag{34}$$

The group shelf-life can be compared to the individual shelf-life (t_{ssl}) based on the shrinkage estimation (28). It is defined as the time period at which the expected fume time curve intersects the 30 days of fume time:

$$30I = X\hat{\beta} + Z\hat{\nu}$$

$$= (I - Z\hat{G}Z'\hat{V}^{-1'}) Z\beta + Z\hat{G}Z'\hat{V}^{-1}y \tag{35}$$

where I is a $n_i \times 1$ vector of 1's and Z is a $n_i \times 1$ vector of $(1, t_{ssl})$.

C. DATA ANALYSIS

Standard linear regression model II based on the reduced data is further investigated for the random effects regression model analysis. As explained earlier, under the AAT, the fume time (y_{ij}) of each lot is repeatedly measured five to nine times in an accelerated condition (65.5 ^{0}C) during the last 20 years. The age (t_{ij}) for each of the lot is computed by subtracting the manufacturing date (see Appendix A) from the date in which when a 45g propellant batch was sent to the heating chamber.

First, in order to estimate the safe shelf-life of a group of similar gun propellants based on the random effects regression model, the within-lot and between-lot models were combined to be a mixed effects model (14). Since the G and R (hence V) are unknown (15), the estimators of G and R can be obtained by a restricted maximum likelihood (REML) (25):

$$\hat{G} = V\hat{a}r(\nu) = \begin{bmatrix} \hat{\sigma}_0^2 & \hat{\sigma}_{01}^2 \\ \hat{\sigma}_{01}^2 & \hat{\sigma}_1^2 \end{bmatrix} = \begin{bmatrix} 0.07079100 & -0.00450653 \\ -0.00450653 & 0.00030661 \end{bmatrix}$$

and $\hat{R} = V\hat{a}r(e) = 0.05926819$. Unknown parameters β and υ can in turn be estimated by Henderson's mixed equation (26) : (1) $\hat{\beta} = [\hat{\gamma}_0, \hat{\gamma}_1]' = [3.81021477, -0.14105394]'$ where $se[\hat{\gamma}_0] = [0.07176290]$ and $se[\hat{\gamma}_1] = [0.00534921]$; (2) $\hat{\nu} = [\hat{\delta}_{01}, \hat{\delta}_{11}, \ldots \hat{\delta}_{0N}, \hat{\delta}_{1N}]'$ and the corresponding standard errors are shown in Table 9.

PARAMETER	SUBJECT	ESTIMATE	STD ERROR
INTERCEPT	LOTID 11142	-0.06196541	0.19329612
AGE	LOTID 11142	0.00479259	0.01346718
INTERCEPT	LOTID 11143	-0.11611040	0.18778713
AGE	LOTID 11143	0.01007674	0.01290832
INTERCEPT	LOTID 11144	-0.16769671	0.19020538
AGE	LOTID 11144	0.01415533	0.01316629
INTERCEPT	LOTID 11145	-0.09516341	0.19031856
AGE	LOTID 11145	0.00494655	0.01335297
INTERCEPT	LOTID 11146	-0.08786149	0.18872403
AGE	LOTID 11146	0.00599071	0.01323274
INTERCEPT	LOTID 11147	-0.21248452	0.18231704
AGE	LOTID 11147	0.01167817	0.01275956
INTERCEPT	LOTID 11148	-0.11397421	0.18758544
AGE	LOTID 11148	0.00853627	0.01298120
INTERCEPT	LOTID 11149	-0.07768438	0.18631350
AGE	LOTID 11149	-0.00123725	0.01287421
INTERCEPT	LOTID 11150	-0.15424392	0.18762834
AGE	LOTID 11150	0.00950736	0.01304139
INTERCEPT	LOTID 11151	-0.12749461	0.18679008
AGE	LOTID 11151	0.00861269	0.01295106
INTERCEPT	LOTID 11156	-0.15799910	0.18658553
AGE	LOTID 11156	0.00818934	0.01299146
INTERCEPT	LOTID 11157	-0.17588616	0.18545442
AGE	LOTID 11157	0.01418657	0.01281651
INTERCEPT	LOTID 11158	-0.13427054	0.18506946
AGE	LOTID 11158	0.00509910	0.01293535
INTERCEPT	LOTID 11159	-0.23813672	0.18472938
AGE	LOTID 11159	0.01251881	0.01296406
INTERCEPT	LOTID 11160	-0.22564272	0.18029667
AGE	LOTID 11160	0.01415942	0.01265016
INTERCEPT	LOTID 11181	-0.16269825	0.18241499
AGE	LOTID 11181	0.00759863	0.01279735
INTERCEPT	LOTID 11182	-0.14882165	0.18459049
AGE	LOTID 11182	0.01126227	0.01279876
INTERCEPT	LOTID 11183	0.17988730	0.18660005
AGE	LOTID 11183	-0.01074349	0.01305771
INTERCEPT	LOTID 11184	0.21035950	0.18618665
AGE	LOTID 11184	-0.01320753	0.01303212
INTERCEPT	LOTID 11185	0.44493213	0.19631611
AGE	LOTID 11185	-0.02624763	0.01352684
INTERCEPT	LOTID 11188	0.24783741	0.18929810
AGE	LOTID 11188	-0.01470002	0.01319538
INTERCEPT	LOTID 11189	0.22284679	0.18633335
AGE	LOTID 11189	-0.01325530	0.01303528
INTERCEPT	LOTID 11190	0.18150475	0.18692804
AGE	LOTID 11190	-0.01253802	0.01310098
INTERCEPT	LOTID 11191	0.22307183	0.18628924
AGE	LOTID 11191	-0.01355682	0.01303573
INTERCEPT	LOTID 11192	0.20898367	0.18614745

AGE	LOTID 11192	-0.01170315	0.01302592
INTERCEPT	LOTID 11193	0.19615700	0.18504953
AGE	LOTID 11193	-0.01181337	0.01297891
INTERCEPT	LOTID 11194	0.17762001	0.18452648
AGE	LOTID 11194	-0.01152574	0.01310043
INTERCEPT	LOTID 11195	0.16493381	0.18380045
AGE	LOTID 11195	-0.01078221	0.01291217

Table 9. $\hat{\nu}$ And Std Error of $\hat{\nu}$

Subsequently, the safe shelf-life of the group of similar lots (t_{sl}) that takes into account the lot-to-lot variation can be obtained as follows:

$$30 = (1, t_{sl})\begin{pmatrix} 3.81021477 \\ -0.14105394 \end{pmatrix} - t(0.95, N\text{-}2)\, SEPRED_{(group)}\ (\tilde{y} \mid t_{sl}) \tag{36}$$

Figure C.22 shows the resulting shelf-life is 8405.12 days (23.02 years).

This result can be compared to the one obtain by the current method (5), which is 8469 days (23.20 years). Note that the current method without taking into account the potential lot-to-lot variation provides a liberal shelf-life that is 64 days longer than the random effects regression model.

Finally, individual shelf-life of lot i (t_{ssl}) based on the shrinkage estimation (28) is computed by checking the time period at which the expected fume time curve intersects the 30 days of fume time (see Table 10). The resulting individual shelf-life (t_{ssl}) varies from 27.998 to 24.942 years. They all exceed the group shelf-life based on either the fixed effects (23.20 years) or the random effects (23.02 years) regression model.

The group shelf-lives based on the prediction limit for the fume time can be compared to those based on the confidence limit for the expected value. They turn out to be 25.66 and

OBS	LOTID	shelf-life (shrinkage estimation)	shelf-life (confidence limit)	shelf-life (prediction limit)
1	11142	26.914	24.520	23.427
2	11143	27.576	25.131	23.971
3	11144	28.056	25.411	24.315
4	11145	26.691	24.365	23.219
5	11146	26.951	24.726	23.424
6	11147	27.173	24.729	23.561
7	11148	27.272	24.863	23.698
8	11149	25.654	23.630	22.459
9	11150	27.167	24.726	23.561
10	11151	27.185	24.794	23.629
11	11156	27.074	24.589	23.356
12	11157	27.998	25.411	24.246
13	11158	26.433	24.178	23.013
14	11159	27.151	24.522	23.492
15	11160	27.600	25.069	23.904
16	11181	26.715	24.383	23.218
17	11182	27.576	25.068	23.972
18	11183	25.563	23.767	22.663
19	11184	25.534	23.629	22.552
20	11185	24.942	23.212	22.191
21	11188	25.526	23.631	22.573
22	11189	25.603	23.698	22.621
23	11190	25.275	23.493	22.459
24	11191	25.555	23.632	22.578
25	11192	25.772	23.835	22.735
26	11193	25.672	23.771	22.658
27	11194	25.590	23.561	22.550
28	11195	25.639	23.698	22.614

Table 10. Individual Shelf-Life of Propellant Lot

24.314 years, which are longer than their counterparts based on the prediction limit.

The individual shelf-lives can also be calculated by checking the time period when 95% one-sided lower prediction or confidence limits intersect 30 days of fume time. The results are shown in Table 10. About 42% of individual shelf-lives based on the prediction limit turned out to be shorter

than the group shelf-life (23.02 years). The rest are as same as the shrinkage estimation that all exceeds the group shelf-life based on either the fixed and random effects regression model.

All calculations are done by using PROC MIXED and PROC REG of a statistical package SAS [Ref.12].

IV. CONCLUSIONS AND RECOMMENDATIONS

A. CONCLUSIONS

Most gun propellant is stored at depots for a long time before it is used. In storage, the quality of the gun propellant may deteriorate and become unstable. In an attempt to avoid disaster due to use of unstable gun propellant, accurate prediction of the safe shelf-life of gun propellant is necessary. The shelf-life estimation methods used currently for a group of similar gun propellant lots are based on a fixed effects regression model. This does not take into consideration the fact that samples from the same lot are more similar than samples between lots. To capitalize on this lot-to-lot variation when estimating the shelf-life, first, a random effects regression model (RERM) is developed. Secondly, a combined mixed effects model is estimated. The estimated model is then used to predict not only the shelf-life of a group of similar lots but also that of each individual lot of 5"/54 NACO gun propellant stockpile. Finally, results of the data analysis are discussed and recommendations are made.

The claimed shelf-life of the NACO propellant lots provided by NSWC is 35 years. It is interesting to note that when the confidence limit method is used instead of the prediction limit method, the standard linear regression model I provides 35 years as the estimated shelf-life. However, the assumptions for standard linear regression model I were not validated. Therefore, the use of the claimed shelf-life (35 years) is not supported and needs to be amended.

The group shelf-life estimated based on a random effects regression model turns out to be 23.02 years. A fixed effects regression model based on the standard linear regression model II provides a slightly longer shelf-life (23.20 years) than 23.02 years. The individual shelf-lives based on the

shrinkage estimation vary from 27.998 to 24.942 years which all exceed the group shelf-lives obtained from both the fixed and the random effects regression models. It indicates that group shelf-life estimated based on the fixed or the random effects regression models can be relatively conservative compared to the individual shelf-lives. When the group shelf-life (23.02 years) is applied to the maintenance of unstable stockpiles, there is a chance that all individual lots would be discarded even though they are still safe. In view of such opportunity loss, use of individual shelf-life is recommended over the group shelf-life when the management of individual lot is expected to cost less than that of the bulk management.

B. RECOMMENDATIONS

In this thesis, a particular propellant stockpile data set is analyzed. Although the approaches proposed can be applied to any other types of propellant, the model selection may be dictated by the nature of data set.

There is a nonlinear decreasing trend in fume time in some individual lots as the propellant ages. This raises questions about the approaches that use traditional linear least squares analysis of fume time data to predict safe shelf-life. This thesis recommends that the nonlinear regression analysis approach be conducted for some individual propellant lots.

APPENDIX A MANUFACTURING DATE

OBS	LOTID	MANUFACTURING DATE	OBS	LOTID	MANUFACTURING DATE
1	11142	10/16/1972	2	11143	11/06/1972
3	11144	11/07/1972	4	11145	11/28/1972
5	11146	12/07/1972	6	11147	4/01/1973
7	11148	12/27/1972	8	11149	12/27/1972
9	11150	12/27/1972	10	11151	1/24/1973
11	11156	1/24/1973	12	11157	2/15/1973
13	11158	4/01/1973	14	11159	4/01/1973
15	11160	10/18/1973	16	11181	10/19/1973
17	11182	4/01/1973	18	11183	10/24/1973
19	11184	10/25/1973	20	11185	7/26/1973
21	11188	7/29/1973	22	11189	10/31/1973
23	11190	10/31/1973	24	11191	11/01/1973
25	11192	11/06/1973	26	11193	1/14/1974
27	11194	1/14/1974	28	11195	1/14/1974

APPENDIX B COMPUTER PROGRAM LIST

The following computer program is a SAS code.

```
OPTIONS LS=80;

                 /*      MASTER SAMPLES        */
DATA ALL1;
INPUT     OB     ID     INDEX     EAGE     EDTF      EYAGE       EYDTF;
ID=_N_;
CARDS;
           1      1     11142      204      756      0.5589     2.07123
           2      1     11142      960      875      2.6301     2.39726
           3      1     11142     1834     1117      5.0247     3.06027
           4      1     11142     2951     1002      8.0849     2.74521
           5      1     11142     3952      741     10.8274     2.03014
           6      1     11142     4692      688     12.8548     1.88493
           7      1     11142     5380      710     14.7397     1.94521
           8      1     11142     6238      578     17.0904     1.58356
           9      2     11143      184      724      0.5041     1.98356
          10      2     11143      908      837      2.4877     2.29315
          11      2     11143     1744     1083      4.7781     2.96712
          12      2     11143     2827     1058      7.7452     2.89863
          13      2     11143     3884      761     10.6411     2.08493
          14      2     11143     4644      699     12.7233     1.91507
          15      2     11143     5343      727     14.6384     1.99178
          16      2     11143     6218      589     17.0356     1.61370
          17      2     11143     6807      552     18.6493     1.51233
          18      3     11144      276      663      0.7562     1.81644
          19      3     11144      939      833      2.5726     2.28219
          20      3     11144     1771     1077      4.8521     2.95068
          21      3     11144     2848     1043      7.8027     2.85753
          22      3     11144     3890      742     10.6575     2.03288
          23      3     11144     4631      683     12.6877     1.87123
          24      3     11144     5314      685     14.5589     1.87671
          25      3     11144     5998      552     16.4329     1.51233
          26      3     11144     6363      806     17.4329     2.20822
          27      4     11145      913      808      2.5014     2.21370
          28      4     11145     1720     1077      4.7123     2.95068
          29      4     11145     2797     1046      7.6630     2.86575
          30      4     11145     3842      764     10.5260     2.09315
          31      4     11145     4605      683     12.6164     1.87123
          32      4     11145     5288      613     14.4877     1.67945
          33      4     11145     5900      559     16.1644     1.53151
          34      5     11146      152      689      0.4164     1.88767
          35      5     11146      841      834      2.3041     2.28493
          36      5     11146     1674     1045      4.5863     2.86301
          37      5     11146     2719     1106      7.4493     3.03014
          38      5     11146     3824      777     10.4767     2.12877
          39      5     11146     4600      694     12.6027     1.90137
          40      5     11146     5294      717     14.5041     1.96438
          41      5     11146     6010      556     16.4658     1.52329
          42      6     11147        0      661      0.0000     1.81096
          43      6     11147      661      855      1.8110     2.34247
          44      6     11147     1515     1017      4.1507     2.78630
          45      6     11147     2532     1057      6.9370     2.89589
          46      6     11147     3588      786      9.8301     2.15342
          47      6     11147     4373      700     11.9808     1.91781
```

48	6	11147	5073	655	13.8986	1.79452
49	6	11147	5727	560	15.6904	1.53425
50	6	11147	6287	459	17.2247	1.25753
51	7	11148	132	728	0.3616	1.99452
52	7	11148	857	834	2.3479	2.28493
53	7	11148	1690	1077	4.6301	2.95068
54	7	11148	2767	1065	7.5808	2.91781
55	7	11148	3831	761	10.4959	2.08493
56	7	11148	4591	708	12.5781	1.93973
57	7	11148	5299	696	14.5178	1.90685
58	7	11148	5994	528	16.4219	1.44658
59	7	11148	6522	596	17.8685	1.63288
60	8	11149	888	806	2.4329	2.20822
61	8	11149	1693	1046	4.6384	2.86575
62	8	11149	2739	1086	7.5041	2.97534
63	8	11149	3824	737	10.4767	2.01918
64	8	11149	4560	699	12.4932	1.91507
65	8	11149	5259	637	14.4082	1.74521
66	8	11149	5895	19	16.1507	0.05205
67	8	11149	6813	477	18.6658	1.30685
68	9	11150	132	728	0.3616	1.99452
69	9	11150	860	824	2.3562	2.25753
70	9	11150	1683	1063	4.6110	2.91233
71	9	11150	2746	1074	7.5233	2.94247
72	9	11150	3819	728	10.4630	1.99452
73	9	11150	4546	682	12.4548	1.86849
74	9	11150	5228	512	14.3233	1.40274
75	9	11150	5740	702	15.7260	1.92329
76	9	11150	6441	538	17.6466	1.47397
77	10	11151	844	823	2.3123	2.25479
78	10	11151	1666	1056	4.5644	2.89315
79	10	11151	2722	1083	7.4575	2.96712
80	10	11151	3804	747	10.4219	2.04658
81	10	11151	4550	698	12.4658	1.91233
82	10	11151	5248	711	14.3781	1.94795
83	10	11151	5958	523	16.3233	1.43288
84	10	11151	6481	557	17.7562	1.52603
85	11	11156	106	709	0.2904	1.94247
86	11	11156	815	842	2.2329	2.30685
87	11	11156	1656	1038	4.5370	2.84384
88	11	11156	2694	1062	7.3808	2.90959
89	11	11156	3755	765	10.2877	2.09589
90	11	11156	4519	665	12.3808	1.82192
91	11	11156	5184	589	14.2027	1.61370
92	11	11156	5773	592	15.8164	1.62192
93	11	11156	6364	463	17.4356	1.26849
94	12	11157	822	822	2.2521	2.25205
95	12	11157	1643	1084	4.5014	2.96986
96	12	11157	2727	1056	7.4712	2.89315
97	12	11157	3782	746	10.3616	2.04384
98	12	11157	4527	710	12.4027	1.94521
99	12	11157	5237	723	14.3479	1.98082
100	12	11157	6118	563	16.7616	1.54247
101	12	11157	6681	662	18.3041	1.81370
102	13	11158	0	738	0.0000	2.02192
103	13	11158	738	829	2.0219	2.27123
104	13	11158	1566	1092	4.2904	2.99178
105	13	11158	2658	1041	7.2822	2.85205
106	13	11158	3698	746	10.1315	2.04384
107	13	11158	4443	681	12.1726	1.86575
108	13	11158	5124	620	14.0384	1.69863

109	13	11158	5743	448	15.7342	1.22740
110	13	11158	6191	482	16.9616	1.32055
111	14	11159	0	730	0.0000	2.00000
112	14	11159	730	842	2.0000	2.30685
113	14	11159	1571	1116	4.3041	3.05753
114	14	11159	2687	1042	7.3616	2.85479
115	14	11159	3728	550	10.2137	1.50685
116	14	11159	4277	624	11.7178	1.70959
117	14	11159	4901	661	13.4274	1.81096
118	14	11159	5562	666	15.2384	1.82466
119	14	11159	6227	489	17.0603	1.33973
120	15	11160	582	834	1.5945	2.28493
121	15	11160	1415	1085	3.8767	2.97260
122	15	11160	2500	1038	6.8493	2.84384
123	15	11160	3537	745	9.6904	2.04110
124	15	11160	4281	695	11.7288	1.90411
125	15	11160	4976	720	13.6329	1.97260
126	15	11160	5695	573	15.6027	1.56986
127	15	11160	6268	581	17.1726	1.59178
128	16	11181	632	826	1.7315	2.26301
129	16	11181	1457	1177	3.9918	3.22466
130	16	11181	2633	953	7.2137	2.61096
131	16	11181	3586	721	9.8247	1.97534
132	16	11181	4306	680	11.7973	1.86301
133	16	11181	4986	686	13.6603	1.87945
134	16	11181	5671	524	15.5370	1.43562
135	16	11181	6195	488	16.9726	1.33699
136	17	11182	0	741	0.0000	2.03014
137	17	11182	741	841	2.0301	2.30411
138	17	11182	1581	1131	4.3315	3.09863
139	17	11182	2712	1023	7.4301	2.80274
140	17	11182	3734	747	10.2301	2.04658
141	17	11182	4480	691	12.2740	1.89315
142	17	11182	5171	716	14.1671	1.96164
143	17	11182	6008	595	16.4603	1.63014
144	17	11182	6603	600	18.0904	1.64384
145	18	11183	622	840	1.7041	2.30137
146	18	11183	1461	1300	4.0027	3.56164
147	18	11183	2760	1105	7.5616	3.02740
148	18	11183	3865	779	10.5890	2.13425
149	18	11183	4643	734	12.7205	2.01096
150	18	11183	5377	713	14.7315	1.95342
151	18	11183	6089	539	16.6822	1.47671
152	19	11184	586	846	1.6055	2.31781
153	19	11184	1431	1322	3.9205	3.62192
154	19	11184	2752	1112	7.5397	3.04658
155	19	11184	3864	781	10.5863	2.13973
156	19	11184	4644	736	12.7233	2.01644
157	19	11184	5380	692	14.7397	1.89589
158	19	11184	6071	515	16.6329	1.41096
159	20	11185	0	698	0.0000	1.91233
160	20	11185	698	1049	1.9123	2.87397
161	20	11185	1746	1492	4.7836	4.08767
162	20	11185	3237	998	8.8685	2.73425
163	20	11185	4234	784	11.6000	2.14795
164	20	11185	5018	766	13.7479	2.09863
165	20	11185	5783	569	15.8438	1.55890
166	20	11185	6352	579	17.4027	1.58630
167	21	11188	0	684	0.0000	1.87397
168	21	11188	684	854	1.8740	2.33973
169	21	11188	1537	1358	4.2110	3.72055

170	21	11188	2894	1072	7.9288	2.93699
171	21	11188	3966	766	10.8658	2.09863
172	21	11188	4731	743	12.9616	2.03562
173	21	11188	5474	689	14.9973	1.88767
174	21	11188	6162	553	16.8822	1.51507
175	22	11189	597	831	1.6356	2.27671
176	22	11189	1427	1351	3.9096	3.70137
177	22	11189	2777	1085	7.6082	2.97260
178	22	11189	3862	795	10.5808	2.17808
179	22	11189	4656	740	12.7562	2.02740
180	22	11189	5396	695	14.7836	1.90411
181	22	11189	6090	564	16.6849	1.54521
182	23	11190	596	866	1.6329	2.37260
183	23	11190	1461	1316	4.0027	3.60548
184	23	11190	2776	1075	7.6055	2.94521
185	23	11190	3851	798	10.5507	2.18630
186	23	11190	4648	719	12.7342	1.96986
187	23	11190	5367	586	14.7041	1.60548
188	23	11190	5952	540	16.3068	1.47945
189	24	11191	601	825	1.6466	2.26027
190	24	11191	1425	1349	3.9041	3.69589
191	24	11191	2773	1096	7.5973	3.00274
192	24	11191	3869	770	10.6000	2.10959
193	24	11191	4638	757	12.7068	2.07397
194	24	11191	5395	683	14.7808	1.87123
195	24	11191	6077	553	16.6493	1.51507
196	25	11192	598	824	1.6384	2.25753
197	25	11192	1421	1357	3.8932	3.71781
198	25	11192	2777	1069	7.6082	2.92877
199	25	11192	3846	799	10.5370	2.18904
200	25	11192	4644	746	12.7233	2.04384
201	25	11192	5390	700	14.7671	1.91781
202	25	11192	6089	615	16.6822	1.68493
203	26	11193	528	851	1.4466	2.33151
204	26	11193	1378	1361	3.7753	3.72877
205	26	11193	2738	1076	7.5014	2.94795
206	26	11193	3814	764	10.4493	2.09315
207	26	11193	4577	749	12.5397	2.05205
208	26	11193	5326	699	14.5918	1.91507
209	26	11193	6024	585	16.5041	1.60274
210	27	11194	528	825	1.4466	2.26027
211	27	11194	1352	1329	3.7041	3.64110
212	27	11194	2680	1075	7.3425	2.94521
213	27	11194	3755	828	10.2877	2.26849
214	27	11194	4582	742	12.5534	2.03288
215	28	11195	528	823	1.4466	2.25479
216	28	11195	1350	1331	3.6986	3.64658
217	28	11195	2680	1059	7.3425	2.90137
218	28	11195	3739	808	10.2438	2.21370
219	28	11195	4546	753	12.4548	2.06301
220	28	11195	5299	694	14.5178	1.90137
221	28	11195	5992	507	16.4164	1.38904

```
                          /*    FLEET RETURN DATA     */
DATA ALL;
INPUT                OBS  ID  INDEX     MDATE     DTF     AGE       ;
CARD;
                       1   1  11142    72.8795    716    1671.0
                       2   1  11142    72.8795    662    4372.0
                       3   1  11142    72.8795    812    1671.0
```

4	1	11142	72.8795	923	1802.0
5	1	11142	72.8795	965	1802.0
6	1	11142	72.8795	665	361.0
7	1	11142	72.8795	769	2364.0
8	1	11142	72.8795	738	1401.0
9	1	11142	72.8795	716	1671.0
10	1	11142	72.8795	479	1183.0
11	1	11142	72.8795	734	2426.0
12	1	11142	72.8795	785	2749.0
13	1	11142	72.8795	685	2896.0
14	1	11142	72.8795	716	1671.0
15	1	11142	72.8795	718	4372.0
16	1	11142	72.8795	795	1802.0
17	2	11143	72.8521	722	1812.0
18	3	11144	72.9384	737	779.5
19	3	11144	72.9384	662	1427.5
20	3	11144	72.9384	814	1521.5
21	3	11144	72.9384	639	5054.5
22	3	11144	72.9384	638	4350.5
23	4	11145	72.9959	719	2325.5
24	4	11145	72.9959	611	5074.5
25	4	11145	72.9959	723	1358.5
26	4	11145	72.9959	812	1628.5
27	4	11145	72.9959	681	2462.5
28	4	11145	72.9959	648	4329.5
29	4	11145	72.9959	697	2788.5
30	4	11145	72.9959	698	2788.5
31	4	11145	72.9959	682	2810.5
32	4	11145	72.9959	693	2810.5
33	4	11145	72.9959	798	1628.5
34	4	11145	72.9959	623	4329.5
35	4	11145	72.9959	795	1759.5
36	4	11145	72.9959	686	1759.5
37	5	11146	73.0219	614	4360.0
38	5	11146	73.0219	628	4320.0
39	5	11146	73.0219	611	4337.0
40	5	11146	73.0219	618	5024.0
41	5	11146	73.0219	643	2123.0
42	5	11146	73.0219	781	1619.0
43	6	11147	73.3370	847	1016.0
44	6	11147	73.3370	710	2259.0
45	6	11147	73.3370	635	4205.0
46	6	11147	73.3370	614	4205.0
47	6	11147	73.3370	722	1635.0
48	6	11147	73.3370	667	2706.0
49	6	11147	73.3370	673	2706.0
50	6	11147	73.3370	613	4950.0
51	6	11147	73.3370	525	2139.0
52	6	11147	73.3370	623	2008.0
53	6	11147	73.3370	677	2197.0
54	7	11148	73.0767	611	5004.0
55	7	11148	73.0767	703	2824.0
56	7	11148	73.0767	728	2354.0
57	7	11148	73.0767	626	5004.0
58	7	11148	73.0767	736	1730.0
59	7	11148	73.0767	1965	1730.0
60	7	11148	73.0767	575	2234.0
61	7	11148	73.0767	667	2801.0
62	7	11148	73.0767	673	2801.0
63	7	11148	73.0767	683	2801.0
64	7	11148	73.0767	690	2801.0

65	7	11148	73.0767	707	2801.0
66	7	11148	73.0767	809	1599.0
67	7	11148	73.0767	681	2433.0
68	7	11148	73.0767	645	4300.0
69	7	11148	73.0767	703	2824.0
70	8	11149	73.0767	791	1599.0
71	8	11149	73.0767	628	4300.0
72	8	11149	73.0767	638	4300.0
73	8	11149	73.0767	611	5004.0
74	8	11149	73.0767	606	5045.0
75	9	11150	73.0767	667	2801.0
76	9	11150	73.0767	673	2801.0
77	9	11150	73.0767	686	2801.0
78	9	11150	73.0767	606	5045.0
79	9	11150	73.0767	594	2234.0
80	9	11150	73.0767	628	4300.0
81	9	11150	73.0767	634	2103.0
82	9	11150	73.0767	809	1599.0
83	9	11150	73.0767	635	4300.0
84	10	11151	73.1493	612	4977.5
85	10	11151	73.1493	611	4290.5
86	10	11151	73.1493	638	4273.5
87	10	11151	73.1493	896	751.5
88	10	11151	73.1493	760	2650.5
89	10	11151	73.1493	797	1444.5
90	10	11151	73.1493	766	2671.5
91	10	11151	73.1493	615	5018.5
92	10	11151	73.1493	860	943.5
93	10	11151	73.1493	692	2523.5
94	11	11156	73.1493	678	4160.5
95	11	11156	73.1493	684	4160.5
96	11	11156	73.1493	590	5018.5
97	11	11156	73.1493	604	4290.5
98	11	11156	73.1493	697	1302.5
99	11	11156	73.1493	615	5018.5
100	12	11157	73.2082	613	4997.0
101	12	11157	73.2082	610	4956.0
102	12	11157	73.2082	623	4252.0
103	12	11157	73.2082	782	1907.0
104	12	11157	73.2082	638	4252.0
105	12	11157	73.2082	627	2055.0
106	12	11157	73.2082	667	2378.0
107	12	11157	73.2082	872	971.0
108	12	11157	73.2082	806	681.0
109	13	11158	73.3370	624	4909.0
110	13	11158	73.3370	697	2664.0
111	13	11158	73.3370	693	2686.0
112	13	11158	73.3370	707	2686.0
113	13	11158	73.3370	627	4222.0
114	13	11158	73.3370	675	2338.0
115	13	11158	73.3370	649	4205.0
116	13	11158	73.3370	728	1635.0
117	13	11158	73.3370	615	4950.0
118	14	11159	73.3370	612	4909.0
119	14	11159	73.3370	616	4950.0
120	14	11159	73.3370	930	924.0
121	15	11160	73.8849	744	434.0
122	15	11160	73.8849	728	1435.0
123	15	11160	73.8849	672	1082.0
124	15	11160	73.8849	677	4005.0
125	15	11160	73.8849	690	4005.0

126	15	11160	73.8849	930	724.0
127	16	11181	73.8877	634	4708.0
128	16	11181	73.8877	672	1081.0
129	16	11181	73.8877	742	1434.0
130	17	11182	73.3370	624	4909.0
131	17	11182	73.3370	624	4909.0
132	17	11182	73.3370	624	4909.0
133	17	11182	73.3370	627	4222.0
134	17	11182	73.3370	930	924.0
135	17	11182	73.3370	707	2664.0
136	17	11182	73.3370	711	2664.0
137	17	11182	73.3370	715	2664.0
138	17	11182	73.3370	706	2686.0
139	18	11183	73.9014	808	428.0
140	18	11183	73.9014	682	3999.0
141	18	11183	73.9014	693	1076.0
142	18	11183	73.9014	668	3999.0
143	18	11183	73.9014	623	4744.0
144	18	11183	73.9014	993	718.0
145	19	11184	73.9041	838	427.0
146	19	11184	73.9041	943	717.0
147	20	11185	73.6562	818	517.5
148	20	11185	73.6562	657	4792.5
149	20	11185	73.6562	703	2214.5
150	20	11185	73.6562	719	1165.5
151	21	11188	73.6644	662	4085.5
152	21	11188	73.6644	694	1162.5
153	21	11188	73.6644	716	1384.5
154	21	11188	73.6644	662	4085.5
155	21	11188	73.6644	781	2019.5
156	22	11189	73.9178	624	4697.0
157	22	11189	73.9178	706	2126.0
158	22	11189	73.9178	662	3993.0
159	22	11189	73.9178	618	4697.0
160	22	11189	73.9178	819	1292.0
161	22	11189	73.9178	829	1160.0
162	22	11189	73.9178	685	1927.0
163	22	11189	73.9178	792	1989.0
164	22	11189	73.9178	716	1292.0
165	22	11189	73.9178	677	3993.0
166	23	11190	73.9205	818	1422.0
167	23	11190	73.9205	791	1647.0
168	23	11190	73.9205	709	2493.0
169	23	11190	73.9205	690	3992.0
170	23	11190	73.9205	869	1422.0
171	24	11191	73.9219	1336	1794.5
172	24	11191	73.9219	716	1290.5
173	24	11191	73.9219	648	3991.5
174	24	11191	73.9219	877	1421.5
175	24	11191	73.9219	925	1421.5
176	24	11191	73.9219	708	2450.5
177	24	11191	73.9219	709	2450.5
178	24	11191	73.9219	707	2472.5
179	24	11191	73.9219	594	4695.5
180	24	11191	73.9219	690	2124.5
181	24	11191	73.9219	653	3991.5
182	25	11192	73.9356	638	4690.5
183	25	11192	73.9356	683	1063.5
184	25	11192	73.9356	700	3873.5
185	25	11192	73.9356	706	3873.5
186	26	11193	74.1219	706	3805.5

```
                    187  26  11193    74.1219    702     2399.5
                    188  26  11193    74.1219    703     2399.5
                    189  26  11193    74.1219    710     2399.5
                    190  26  11193    74.1219    726     2399.5
                    191  26  11193    74.1219    918      637.5
                    192  26  11193    74.1219    711     2399.5
                    193  26  11193    74.1219    725     2399.5
                    194  26  11193    74.1219    726     2399.5
                    195  26  11193    74.1219    731     2399.5
                    196  26  11193    74.1219    697     2044.5
                    197  26  11193    74.1219    700     2051.5
                    198  26  11193    74.1219    716     1217.5
                    199  26  11193    74.1219    654     3918.5
                    200  26  11193    74.1219    646     3918.5
                    201  27  11194    74.1219    647     4622.5
                    202  27  11194    74.1219    688     2044.5
                    203  28  11195    74.1219    612     4622.5
                    204  28  11195    74.1219    609     4622.5
;
PROC SORT; BY INDEX;
PROC PRINT DATA= ALL1;VAR INDEX EAGE EDTF EYAGE EYDTF;

         /*     COMBINE MASTER SAMPLE AND FLEET RETURN DATA      */
DATA NEW;
MERGE ALL1 ALL2 ;  BY INDEX;
IF ID = LAG(ID) THEN EAGE = .;
IF ID = LAG(ID) THEN EDTF = .;
PROC PRINT DATA=NEW; VAR INDEX MDATE DTF AGE EDTF EAGE;
PROC UNIVARIATE;
PROC PLOT;
     PLOT DTF*AGE='A' EDTF*EAGE='*';
PROC PLOT;
     PLOT DTF*AGE='A' EDTF*EAGE='*'/OVERLAY;

          /*     STANDARD LINEAR REGRESSION MODEL I        */

PROC REG ; MODEL DTF = AGE ;

         /*     STANDARD LINEAR REGRESSION MODEL II        */
         /*         ( DELET EAGE BELOW 1100 DAYS )         */


DATA ALL3(DROP= OB ID EYAGE  EYDTF);SET ALL1;

DATA ALL4;SET ALL3;
IF EAGE LE 1100 THEN DELETE;
PROC PLOT ;
     PLOT EDTF*EAGE='*';
PROC REG ; MODEL EDTF= EAGE;
OUTPUT OUT=OUT1 P= PRED  R=RES;
PROC PRINT;

DATA ALL5 (DROP= RES ) ;MERGE ALL4 OUT1; BY INDEX;
PROC PRINT;

DATA ALL6(DROP=OBS);  SET ALL5;BY INDEX;

W=(EAGE-4146.15)*(EAGE-4146.15);
SXX= 2521553*(180-1);
SEPRED = 98.295 * SQRT(1+(1/180)+(W/SXX));
L95=PRED-1.645*SEPRED;
```

```
U95=PRED+1.645*SEPRED;
EAGE30=30;
PROC PRINT;
PROC PLOT ;
     PLOT EDTF*EAGE='A' PRED*EAGE='-' L95*EAGE='.'
          U95*EAGE='.' EAGE30*EAGE='-'/OVERLAY;

     PLOT  PRED*EAGE='*' L95*EAGE='.'
          U95*EAGE='.' EAGE30*EAGE='-'/OVERLAY;
DATA EXTEN1;
DO DAGE = 8000 TO 12000 BY 100;
PRED=1045.510-0.07*EAGE;

W=(EAGE-4146.15)*(EAGE-4146.15);
SXX= 2521553*(180-1);
SEPRED = 98.295 * SQRT(1+(1/180)+(W/SXX));
L95=PRED-1.645*SEPRED;
U95=PRED+1.645*SEPRED;
OUTPUT;
END;
PROC PRINT DATA=EXTEN1;VAR EAGE PRED L95 U95;

             /*     LINEARIZING TRANSFORMATION MODEL I          */
             /*            (TRANSFER EAGE TO 1/EAGE)            */

DATA ALL7 ; SET ALL1;
IF EAGE LE 1100 THEN DELETE;

Y=1/EAGE;
PROC REG ; MODEL EDTF= Y;
OUTPUT OUT=OUT2 P= PRED  R=RES STDI=SEP;

PROC PLOT;
     PLOT EDTF*EAGE;
DATA ALL8 (DROP= RES ) ;MERGE ALL7 OUT2; BY INDEX;
PROC UNIVARIATE;

DATA ALL9 (DROP=OBS);  SET ALL8;BY INDEX;

W=(Y-0.000297)*(Y-0.000297);
SXX= 0.000164*0.000164*(180-1);
SEPRED = 112.854* SQRT(1+(1/180)+(W/SXX));
L95=PRED-1.645*SEPRED;
U95=PRED+1.645*SEPRED;
PROC PRINT DATA=ALL9 ;VAR INDEX EAGE EDTF  PRED U95 L95;
PROC PLOT ;
 PLOT EDTF*Y='A' PRED*EAGE='-' L95*EAGE='.'
          U95*EAGE='.' /OVERLAY;

     PLOT  PRED*EAGE='*' L95*EAGE='.'
          U95*EAGE='.' /OVERLAY;

 DATA EXTEN2;
 DO EAGE=15000 TO 35000 BY 100;
 PRED=411.737+1315827*(1/EAGE);

W=((1/EAGE)-0.000297)*((1/EAGE)-0.000297);
SXX= 0.000164*0.000164*(180-1);
SEPRED = 112.854* SQRT(1+(1/180)+(W/SXX));
L95=PRED-1.645*SEPRED;
U95=PRED+1.645*SEPRED;
```

```
OUTPUT;
END;
PROC PRINT DATA=EXTEN2; VAR EAGE PRED L95 U95;

           /*      LINEARIZING TRANSFORMATION MODEL II          */
           /*         (TRANSFER EDTF TO LOG(EDTF))              */

DATA ALL10; SET ALL1;
IF EAGE LE 1100 THEN DELETE;

EDTFLOG = LOG(EDTF);
PROC REG ; MODEL EDTFLOG = EAGE;
OUTPUT OUT=OUT3 P= PRED  R=RES STDI=SEP;

PROC PLOT;
     PLOT EDTF*EAGE;
DATA ALL11(DROP= RES ) ;MERGE ALL10 OUT3; BY INDEX;
PROC UNIVARIATE;

DATA ALL12(DROP=OBS);  SET ALL11;BY INDEX;

W=(EAGE-4146)*(EAGE-4146);
SXX= 2521553*(180-1);
SEPRED = 0.2742 * SQRT(1+(1/180)+(W/SXX));
L95=PRED-1.645*SEPRED;
U95=PRED+1.645*SEPRED;
PROC PRINT DATA=ALL12 ;VAR INDEX EAGE EDTF PRED L95 U95;
PROC PLOT ;
     PLOT EDTFLOG*EAGE='A' PRED*EAGE='-' L95*EAGE='.'
          U95*EAGE='.' /OVERLAY;

     PLOT  PRED*EAGE='*' L95*EAGE='.'
          U95*EAGE='.' /OVERLAY;

 DATA EXTEN3;
 DO EAGE=14000 TO 20000 BY 100;
 EDTFH=EXP(7.3787-0.000197*EAGE);
 PRED =7.3787-0.000197*EAGE;

W=(EAGE-4146)*(EAGE-4146);
SXX= 2521553*(180-1);
SEPRED = 0.2742 * SQRT(1+(1/180)+(W/SXX));
L95=PRED-1.645*SEPRED;
U95=PRED+1.645*SEPRED;
EXPL95=EXP(PRED-1.645*SEPRED);
EXPU95=EXP(PRED+1.645*SEPRED);
OUTPUT;
END;
PROC PRINT DATA=EXTEN3;VAR EAGE PRED L95 U95 EXPL95;


          /*           FLEET RETURN DATA            */

DATA ALL14 ;SET ALL2;
IF DTF GE  1200 THEN DELETE;

PROC PLOT;
     PLOT DTF*AGE='*';
PROC REG ; MODEL DTF= AGE;
OUTPUT OUT=OUT5 P= PRED  R=RES STDI=SEP;
```

```
PROC PRINT;
PROC UNIVARIATE;
DATA ALL15(DROP= RES ) ;MERGE ALL14 OUT5; BY INDEX;
PROC PRINT DATA = ALL15; VAR INDEX DTF AGE PRED;
PROC PRINT ;


DATA ALL16;  SET ALL15;BY INDEX;
W=(AGE-2821.968)*(AGE-2821.968);


SXX= 1928683*(202-1);
SEPRED = 61.32 * SQRT(1+(1/202)+(W/SXX));
L95=PRED-1.645*SEPRED;
U95=PRED+1.645*SEPRED;
AGE30=30;
PROC PRINT; VAR INDEX AGE DTF PRED U95 L95;
PROC PLOT ;
   PLOT DTF*AGE='A' PRED*AGE='-' L95*AGE='.'
          U95*AGE='.' AGE30*AGE='-'/OVERLAY;

     PLOT  PRED*AGE='*' L95*AGE='.'
          U95*AGE='.' AGE30*AGE='-'/OVERLAY;


DATA EXTEN5;
DO AGE=10000 TO 16400 BY 100;
PRED= 823.375-0.0429 *AGE;

W=(AGE-2821.968)*(AGE-2821.968);
SXX= 1928683*(202-1);
SEPRED = 61.32 * SQRT(1+(1/202)+(W/SXX));
L95=PRED-1.645*SEPRED;
TEST=PRED-1.699*SEPRED;
U95=PRED+1.645*SEPRED;
AGE30=30;

OUTPUT;
END;

PROC PRINT DATA= EXTEN5;VAR  AGE PRED L95 U95 TEST;
PROC PLOT;
     PLOT L95*AGE='*'   AGE30*AGE='.'/OVERLAY;


             /*         WEIGHTED LEAST SQUARE MODEL    */
             /*            (DTF/AGE VS 1/AGE)          */
DATA ALL20 ;SET ALL2;
IF DTF GE  1200 THEN DELETE;
NEWDTF=DTF/AGE ;
NEWAGE=1/AGE;
PROC REG ; MODEL NEWDTF= NEWAGE;
OUTPUT OUT=OUT21 P= PRED  R=RES STDI=SEP ;
PROC PRINT;
PROC UNIVARIATE;
DATA ALL22;MERGE ALL20 OUT21;BY INDEX;
       U95 = PRED+1.645*SEP;
       L95 = PRED-1.645*SEP;
PROC PRINT DATA=ALL22;VAR AGE DTF PRED L95 U95 SEP;

PROC PLOT;
```

```
     PLOT RES* PRED='*' ;
PROC PLOT;
     PLOT L95 *AGE='*' PRED*AGE='*' U95*AGE='*'/OVERLAY;

           /*       POWER TRANSFORMATION MODEL          */
           /*             (DTF**-3 VS AGE)              */
DATA ALL23 ;SET ALL2;
IF DTF GE  1200 THEN DELETE;
NEWDTF=DTF**(-3);
PROC REG ; MODEL NEWDTF= AGE;
OUTPUT OUT=OUT24 P= PRED  R=RES STDI=SEP ;
PROC PRINT;
PROC UNIVARIATE;
DATA ALL25;MERGE ALL23 OUT24;BY INDEX;
   U95 = PRED+1.645*SEP;
   L95 = PRED-1.645*SEP;
PROC PRINT DATA=ALL25;VAR AGE DTF PRED L95 U95 SEP;
PROC PLOT;
     PLOT RES* PRED='*' ;
PROC PLOT;
     PLOT L95 *AGE='*' PRED*AGE='*' U95*AGE='*'/OVERLAY;


DATA F1M4EXT;
DO AGE=900000   TO 1000000   BY 100;
PRED= (1.75*(10**(-9)))+(4.87*(10**(-13))) *AGE;

W=(AGE-2821.968)*(AGE-2821.968);
SXX= 1928683*(202-1);
SEPRED = SQRT(6.204807*(10**(-19))) * SQRT(1+(1/202)+(W/SXX));
U95=(PRED-1.645*SEPRED)**(-1/3);
L95=(PRED+1.645*SEPRED)**(-1/3);
AGE30=30;
OUTPUT;
END;

PROC PRINT DATA= F1M4EXT;VAR  AGE PRED L95 U95 ;
PROC PLOT;
PLOT L95*AGE='*' PRED*AGE='.' U95*AGE='[' AGE30*AGE='.'/OVERLAY;


DATA ONE( DROP=OBS); SET ALL1;
FUME=EAGE/365; AGE=EAGE/365;

PROC PRINT; VAR LOTID AGE FUME;

DATA ONE; SET ALL1; IF AGE < 3 THEN DELETE;


                     /*  WITHIN-LOT MODEL */

PROC MIXED;
CLASS LOTID;
MODEL FUME = LOTID AGE*LOTID / NOINT S PREDICTED;


                  /*  FIXED EFFECTS REGRESSION MODEL */

PROC MIXED;
CLASS LOTID;
MODEL FUME = AGE /  S PREDICTED;
```

```
                    /* RANDOM EFFECTS REGRESSION MODEL */

PROC MIXED ABSOLUTE;
CLASS LOTID;
MODEL FUME = AGE /   S PREDICTED;
RANDOM   INT    AGE   / CL G   S SUB=LOTID TYPE=UN ;
PROC PRINT;

   /*  COMPUTE PRED SEPRED U95 L95 FOR RANDOM EFFECTS MODEL   */

DATA TWO(DROP= OBS); SET ONE; BY LOTID;
PRED=3.81021477-0.14105394 * AGE;
SEPRED=SQRT(( 0.070791)+(2*AGE*(-0.00450653))
            +(AGE*AGE*0.00030661)+(0.05926819));
U95=PRED+1.645*SEPRED;
L95=PRED-1.645*SEPRED;
PREDD=PRED*365;
U95D=U95*365;
L95D=L95*365;
PROC PRINT; VAR LOTID AGE FUME PRED PREDD U95D L95D;

          /*  GROUP SHELF-LIFE ESTIMATION   */

DATA EXTEN7;
   DO AGE=(5000/365) TO (10000/356) BY (100/365);
      PRED=3.81021477-0.14105394 * AGE;
      SEPRED=SQRT(( 0.070791)+(2*AGE*(-0.00450653))
                  +(AGE*AGE*0.00030661));
      U95=PRED+1.645*SEPRED;
      L95=PRED-1.645*SEPRED;
      PREDD=PRED*365;
      U95D=U95*365;
      L95D=L95*365;
      AGED=AGE*365;
  OUTPUT;
  END;
 PROC PRINT; VAR   AGE   AGED   PRED PREDD L95D      ;

                        /*  SHRINKAGE ESTIMATION   */

DATA THREE; INPUT LOTID INTERCEP SLOPE;
CARDS;
11142   -0.06196541   0.00479259
11143   -0.11611040   0.01007674
11144   -0.16769671   0.01415533
11145   -0.09516341   0.00494655
11146   -0.08786149   0.00599071
11147   -0.21248452   0.01167817
11148   -0.11397421   0.00853627
11149   -0.07768438  -0.00123725
11150   -0.15424392   0.00950736
11151   -0.12749461   0.00861269
11156   -0.15799910   0.00818934
11157   -0.17588616   0.01418657
```

```
11158    -0.13427054    0.00509910
11159    -0.23813672    0.01251881
11160    -0.22564272    0.01415942
11181    -0.16269825    0.00759863
11182    -0.14882165    0.01126227
11183     0.17988730   -0.01074349
11184     0.21035950   -0.01320753
11185     0.44493213   -0.02624763
11188     0.24783741   -0.01470002
11189     0.22284679   -0.01325530
11190     0.18150475   -0.01253802
11191     0.22307183   -0.01355682
11192     0.20898367   -0.01170315
11193     0.19615700   -0.01181337
11194     0.17762001   -0.01152574
11195     0.16493381   -0.01078221
20000     0.00000000    0.00000000
;
PROC SORT; BY LOTID;
PROC PRINT;

DATA FOUR; SET THREE ;
INTERC1=INTERCEP + 3.81021477;
SLOPE1=SLOPE-0.14105394;
PROC PRINT;VAR LOTID INTERC1 SLOPE1;

DATA FIVE; SET THREE ;
INTERC1=INTERCEP + 3.81021477;
SLOPE1=SLOPE-0.14105394;
   DO AGE=(5000/365) TO (15000/356) BY (100/365);
      TSSL = INTERC1 + SLOPE1 * AGE ; BY LOTID;
      TSSLD=TSSL*365;
      AGED= AGE*365;
  OUTPUT;
  END;
 PROC PRINT; VAR  LOTID  AGE  AGED TSSL TSSLD  ;
```

APPENDIX C FIGURES

The figures shown in this appendix are described in Chapter II and IV are as follows :

Figure C.1 -Figure C.3	Standard Linear Regression Model I.
Figure C.4 -Figure C.6	Standard Linear Regression Model II.
Figure C.7 -Figure C.8	Linearizing Transformation Model I.
Figure C.9 -Figure C.11	Linearizing Transformation Model II.
Figure C.12-Figure C.14	Standard Regression Model Based on Fleet return data.
Figure C.15	Weighted Least Square Model.
Figure C.16-Figure C.17	Power Transformation Model.
Figure C.18-Figure C.21	Individual fitted Models.
Figure C.22	Group shelf-life estimation.

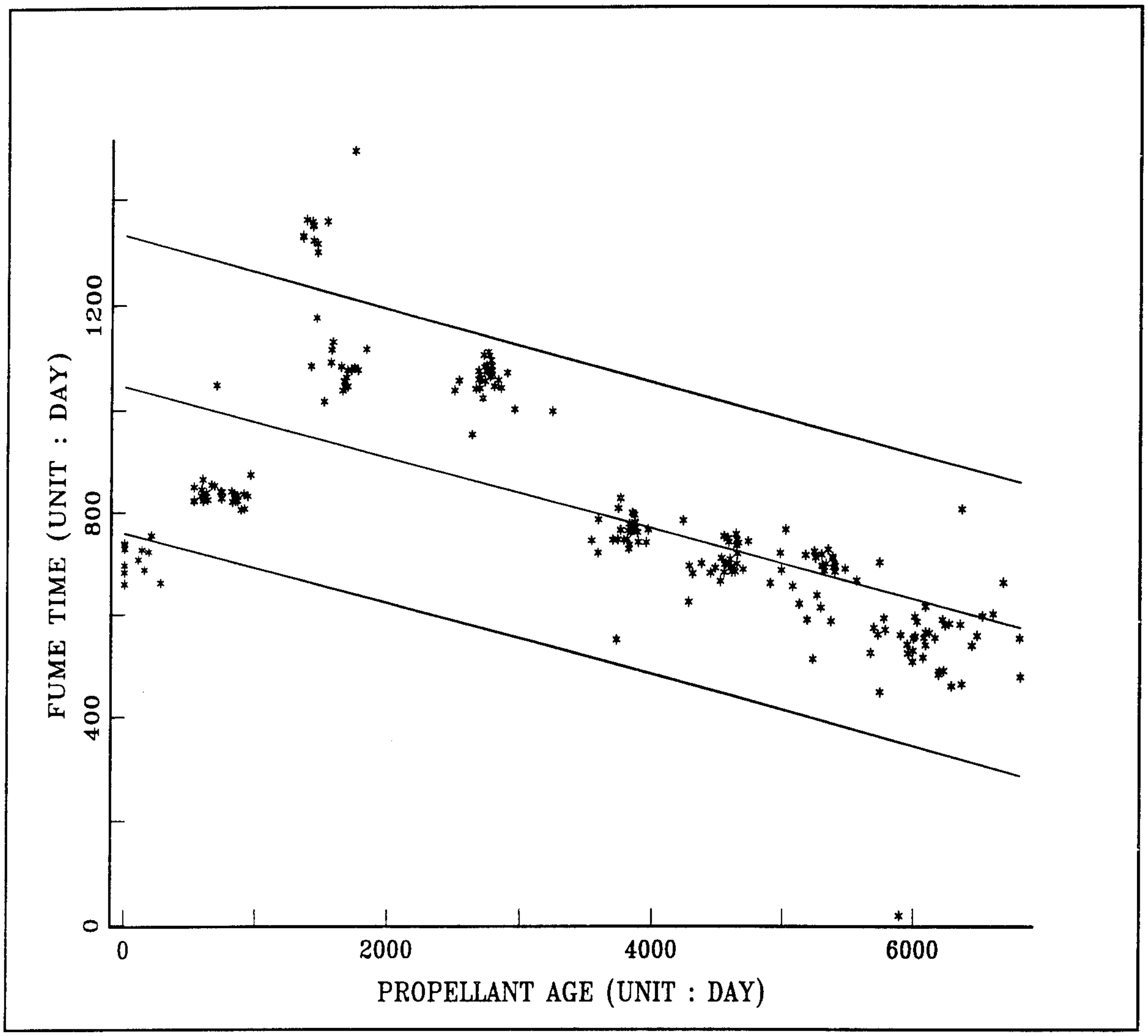

Figure C.1. Fume time vs propellant age based on the standard linear regression model I.

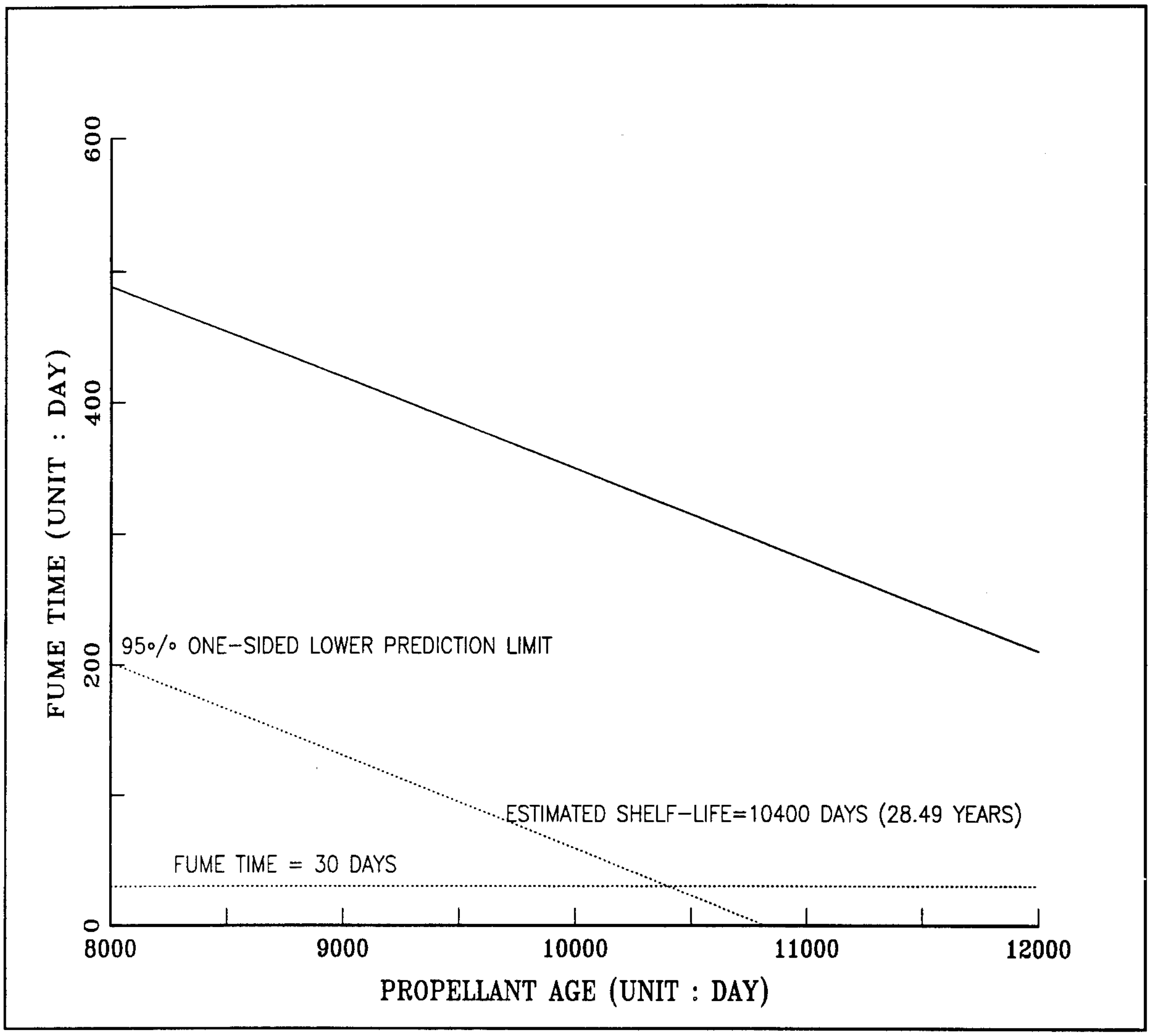

Figure C.2. Group shelf-life estimation based on the standard linear regression model I.

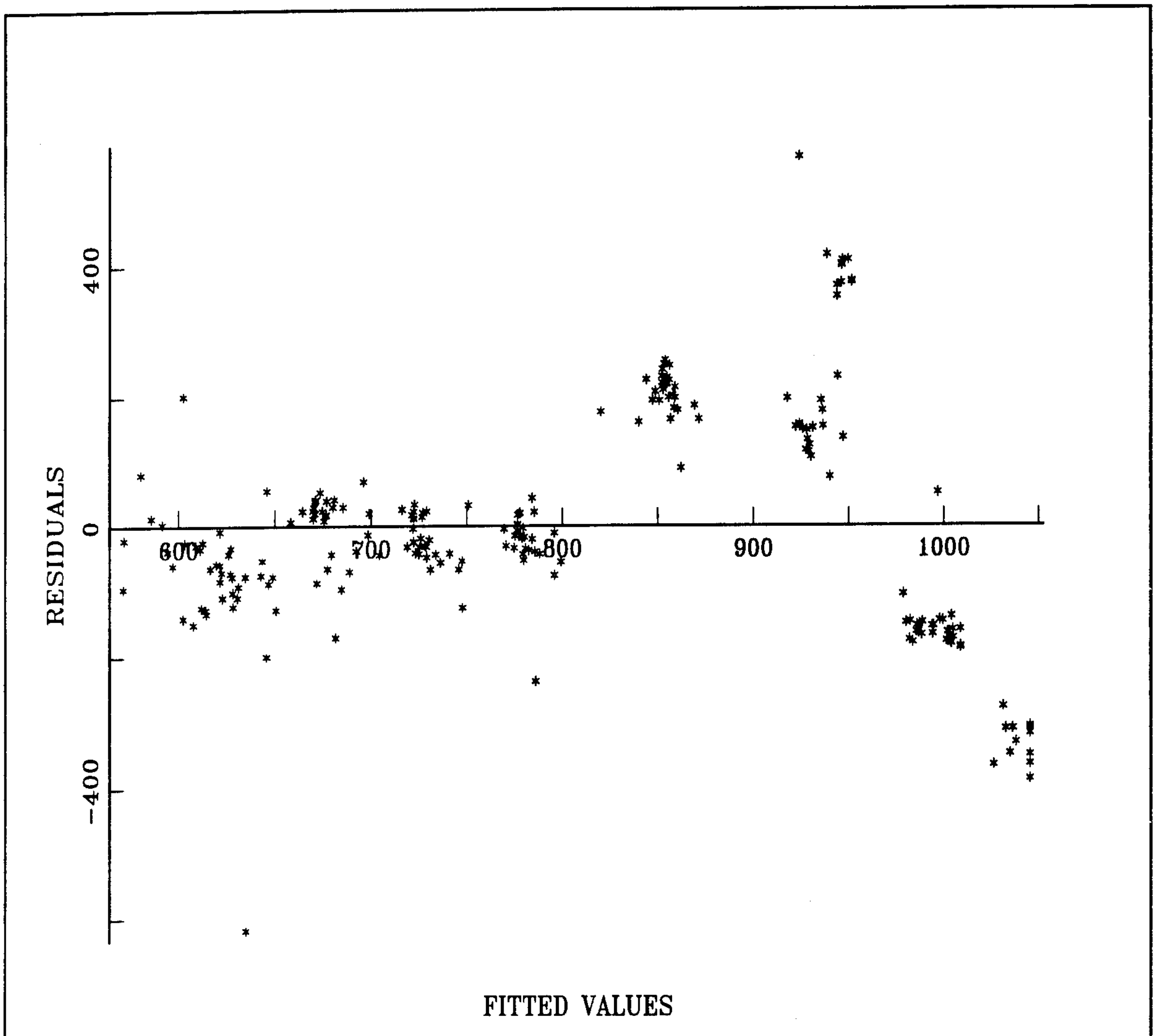

Figure C.3. Residual analysis based on the standard linear regression model I.

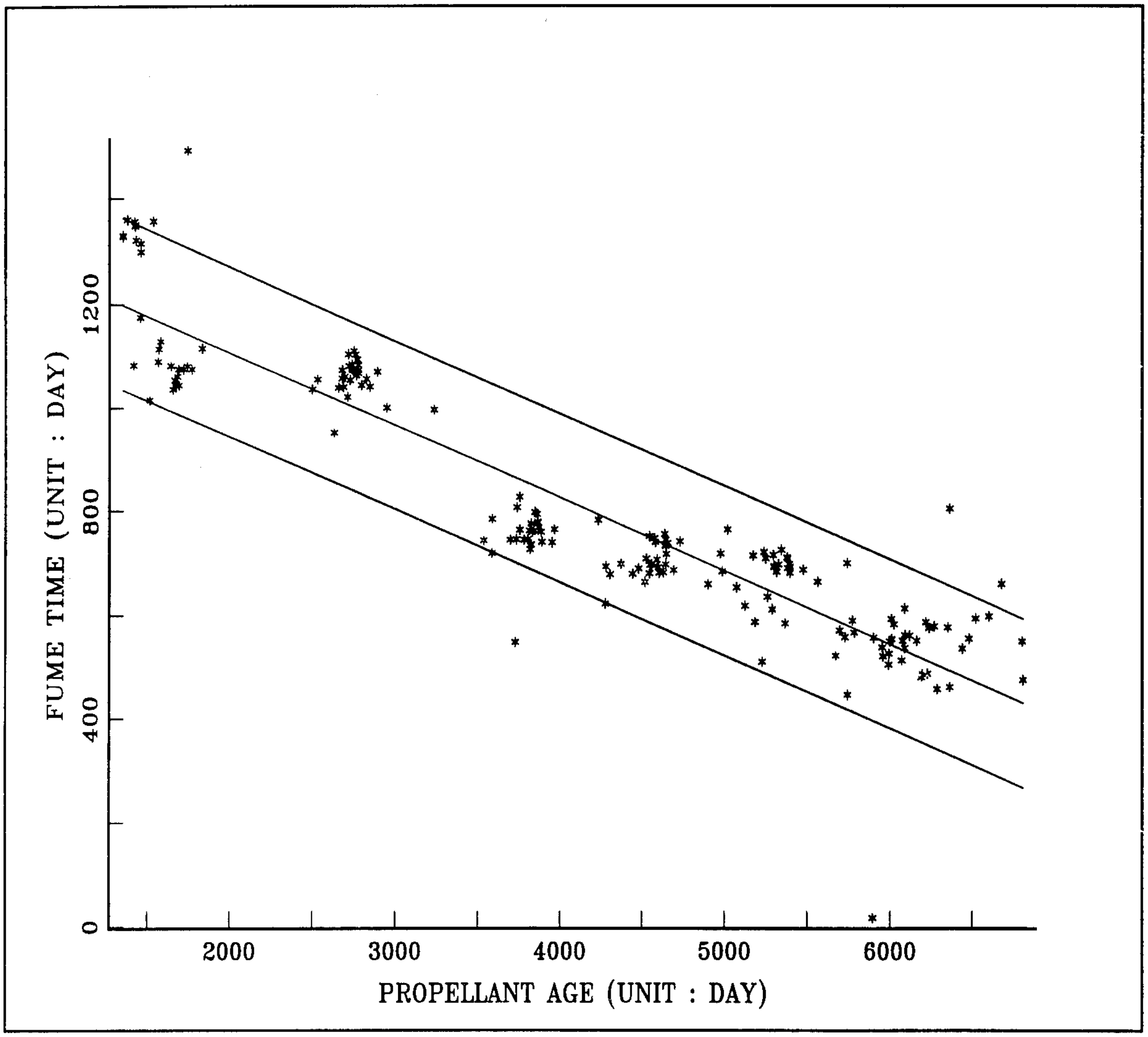

Figure C.4. Fume time vs propellant age based on the standard linear regression model II.

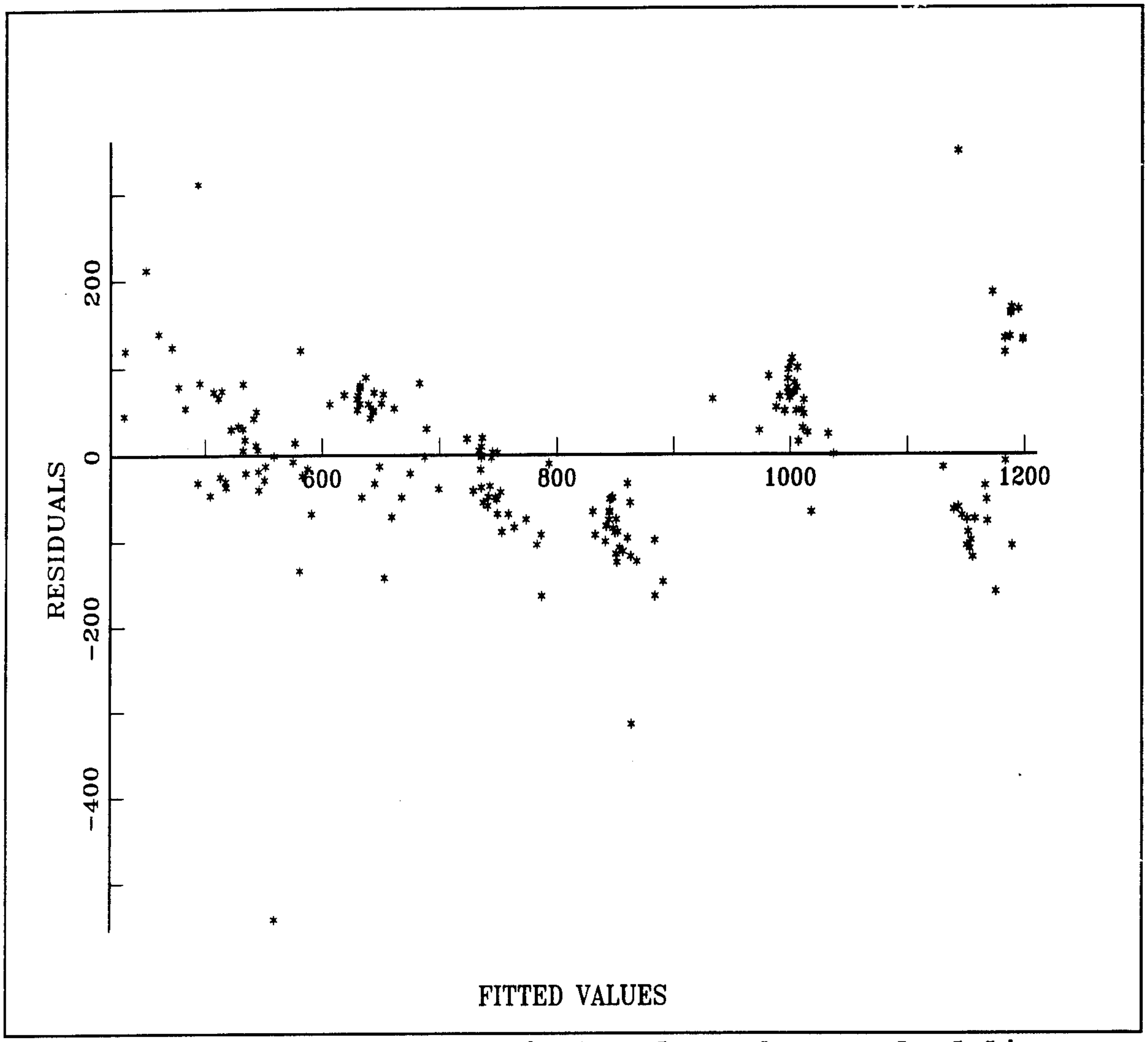

Figure C.5. Residual analysis based on the standard linear regression model II.

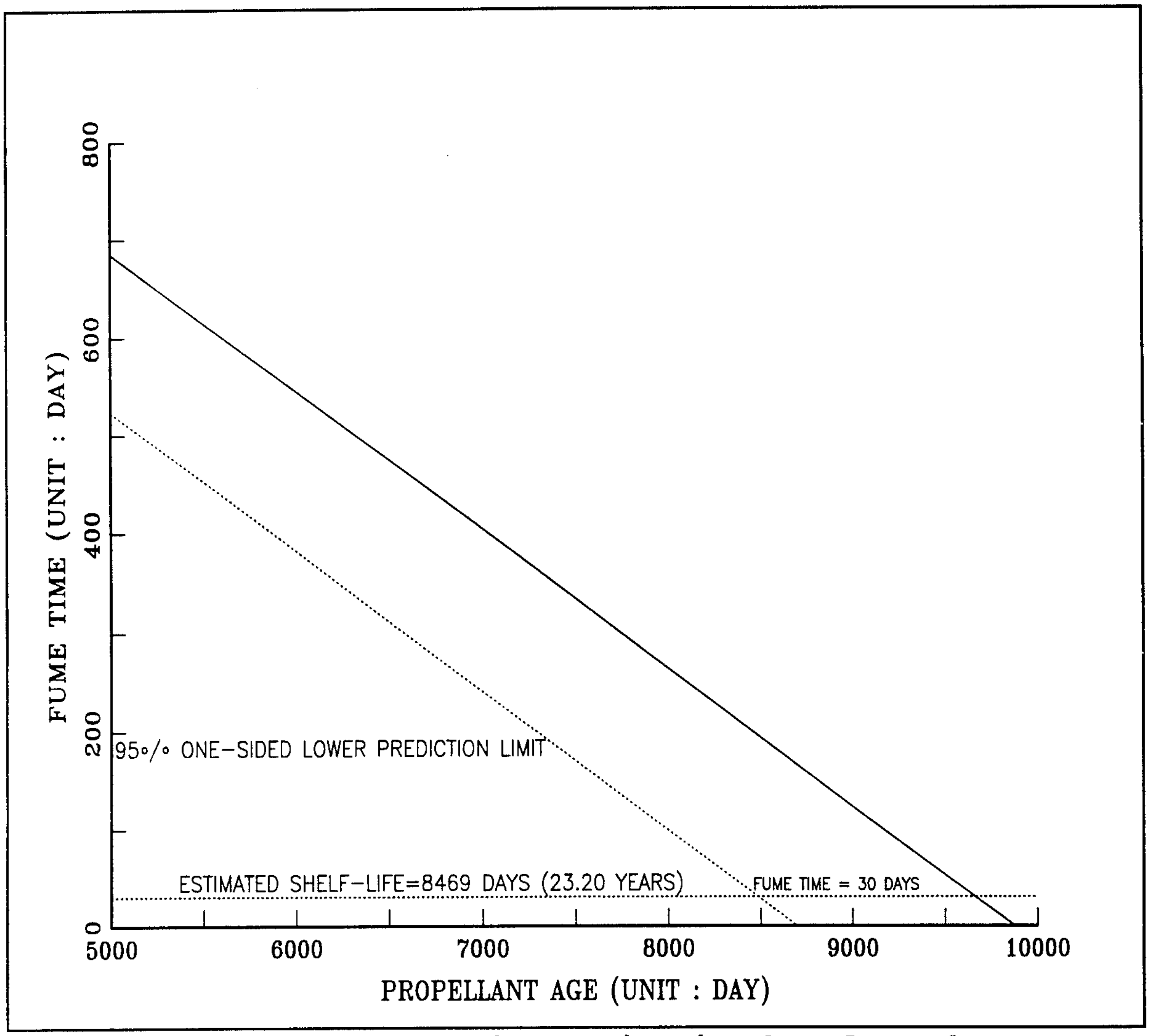

Figure C.6. Group shelf-life estimation based on the standard linear regression model II.

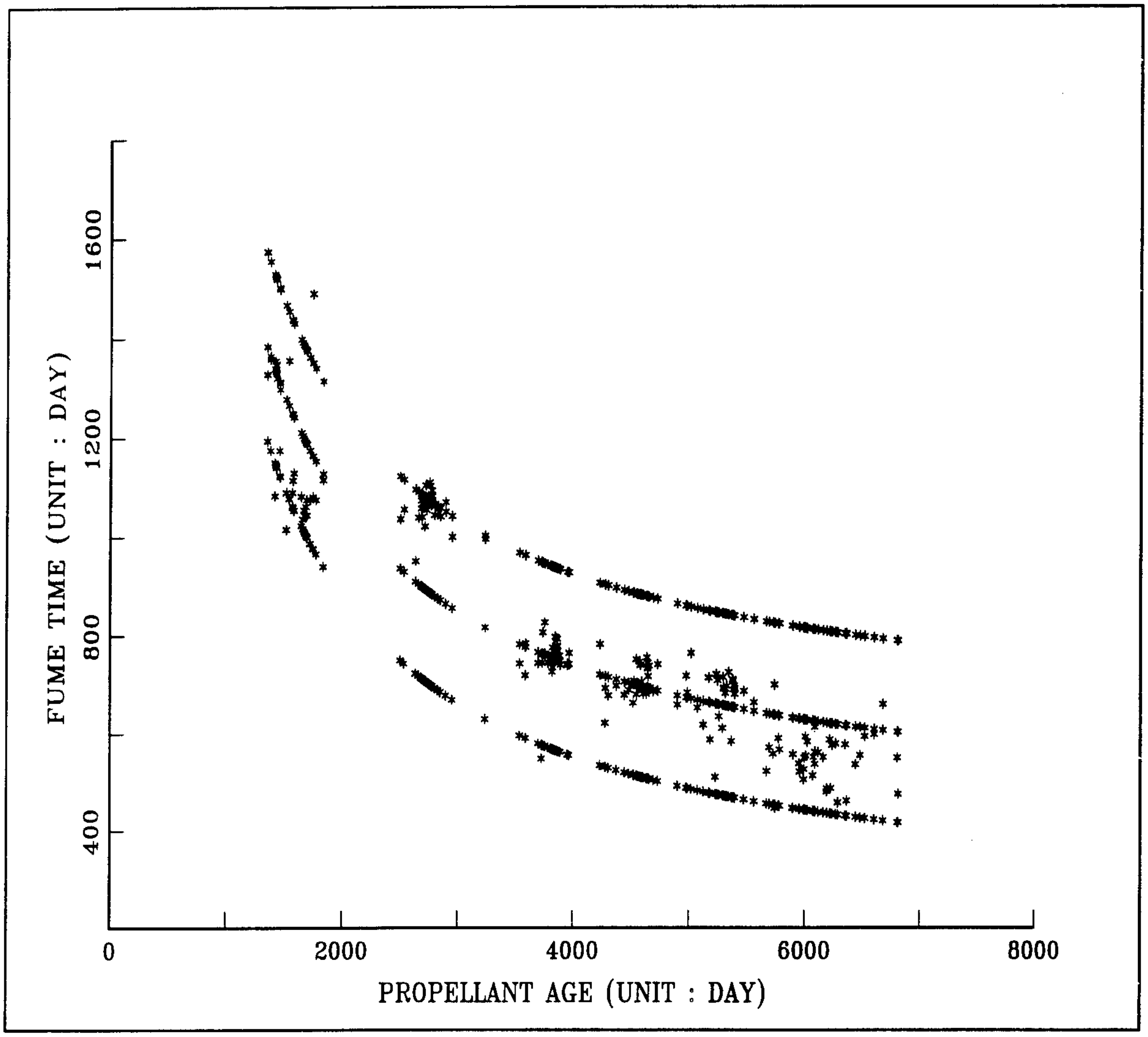

Figure C.7. Fume time vs propellant age based on the linearizing transformation model I.

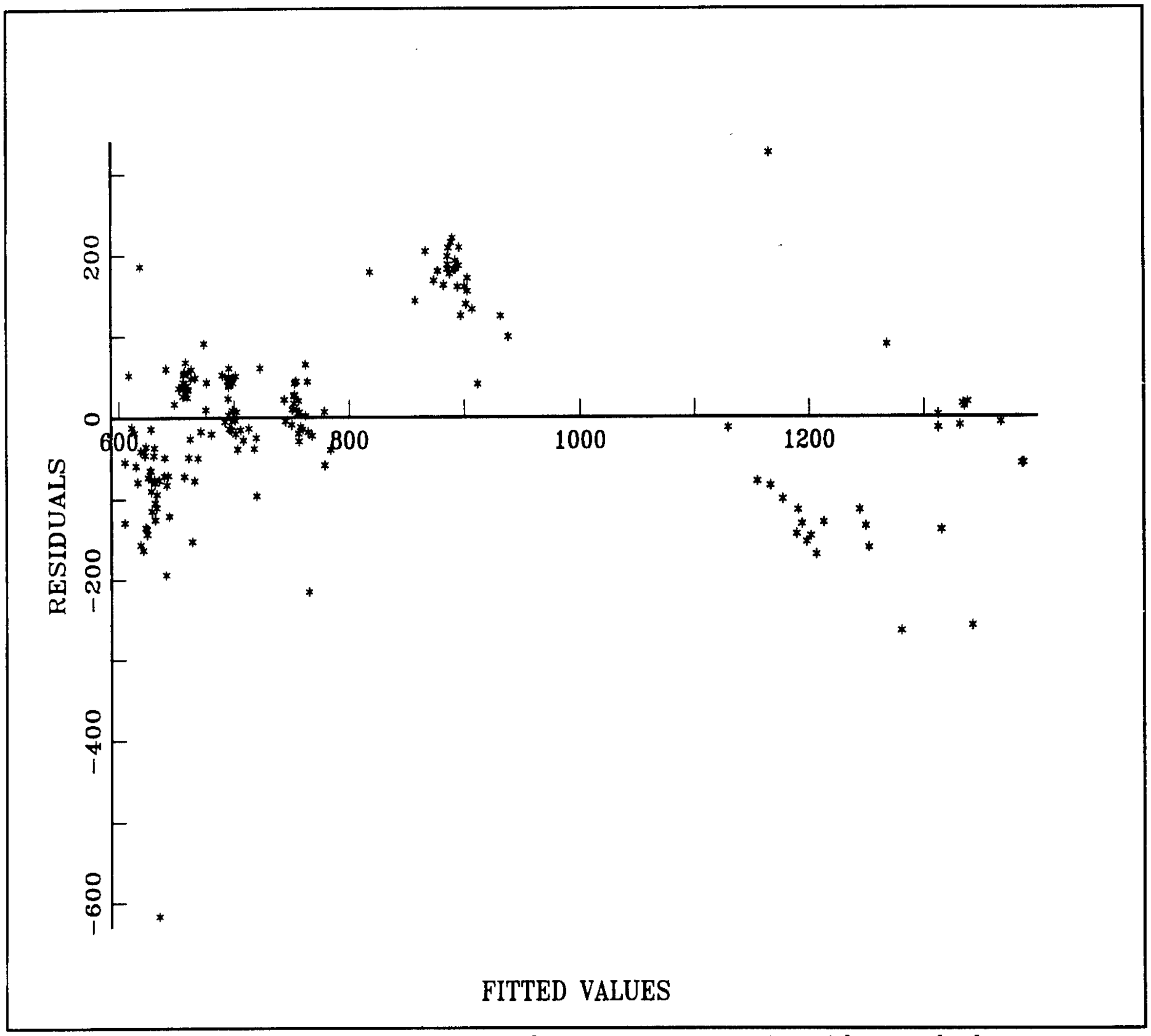

Figure C.8. Residual analysis based on the linearizing transformation model I.

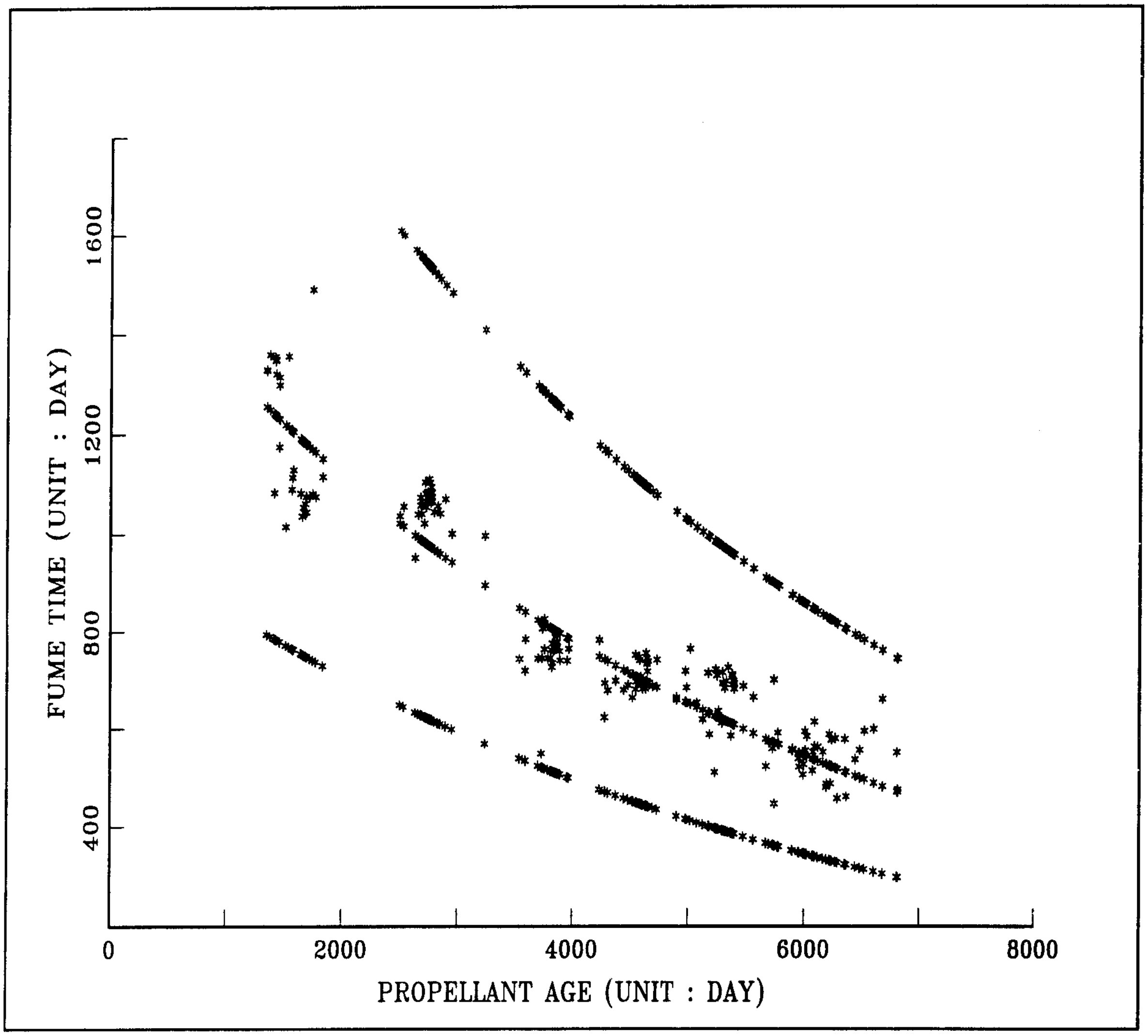

Figure C.9. Fume time vs propellant age based on the linearizing transformation model II.

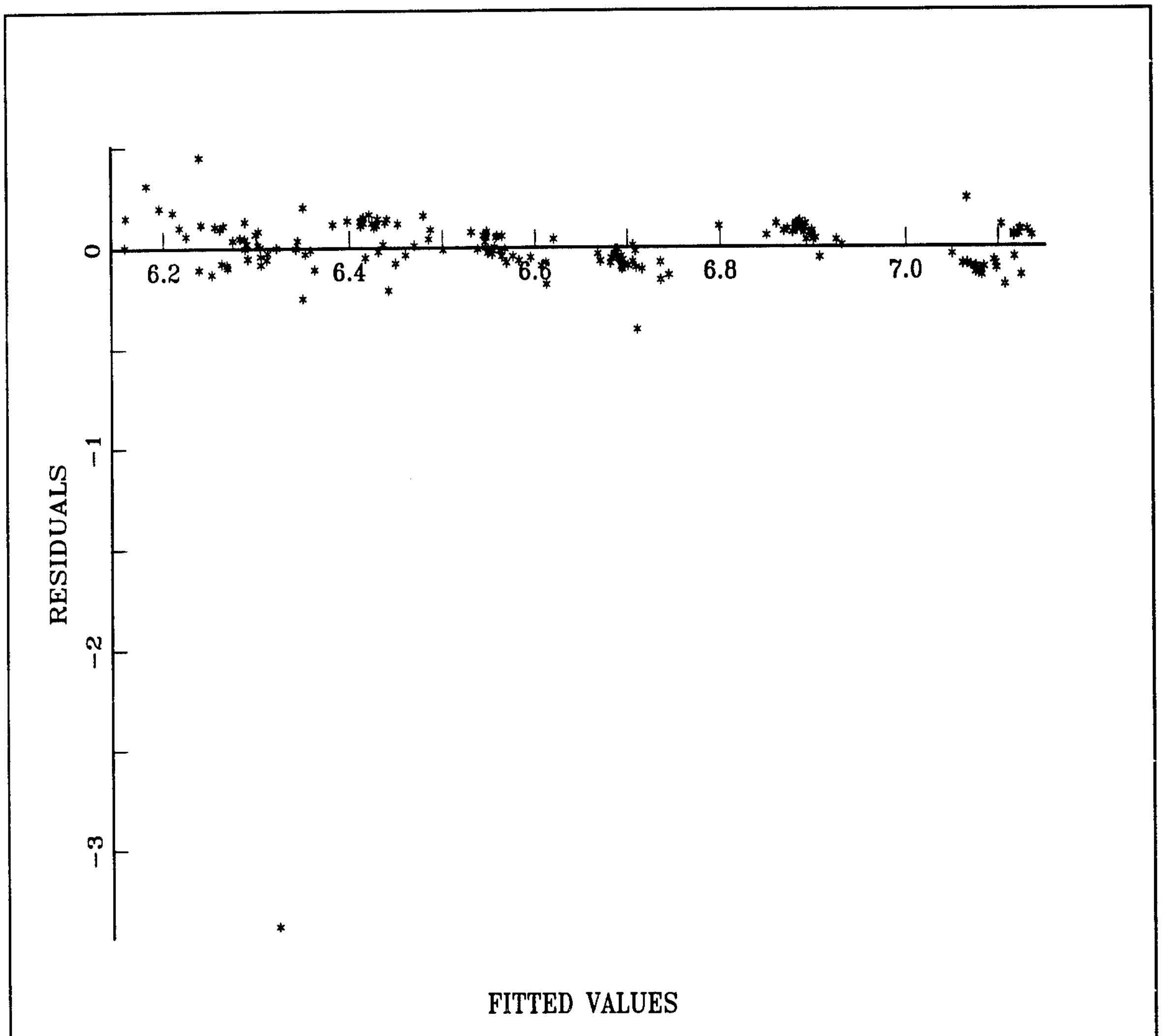

Figure C.10. Residual analysis based on the linearizing transformation model II.

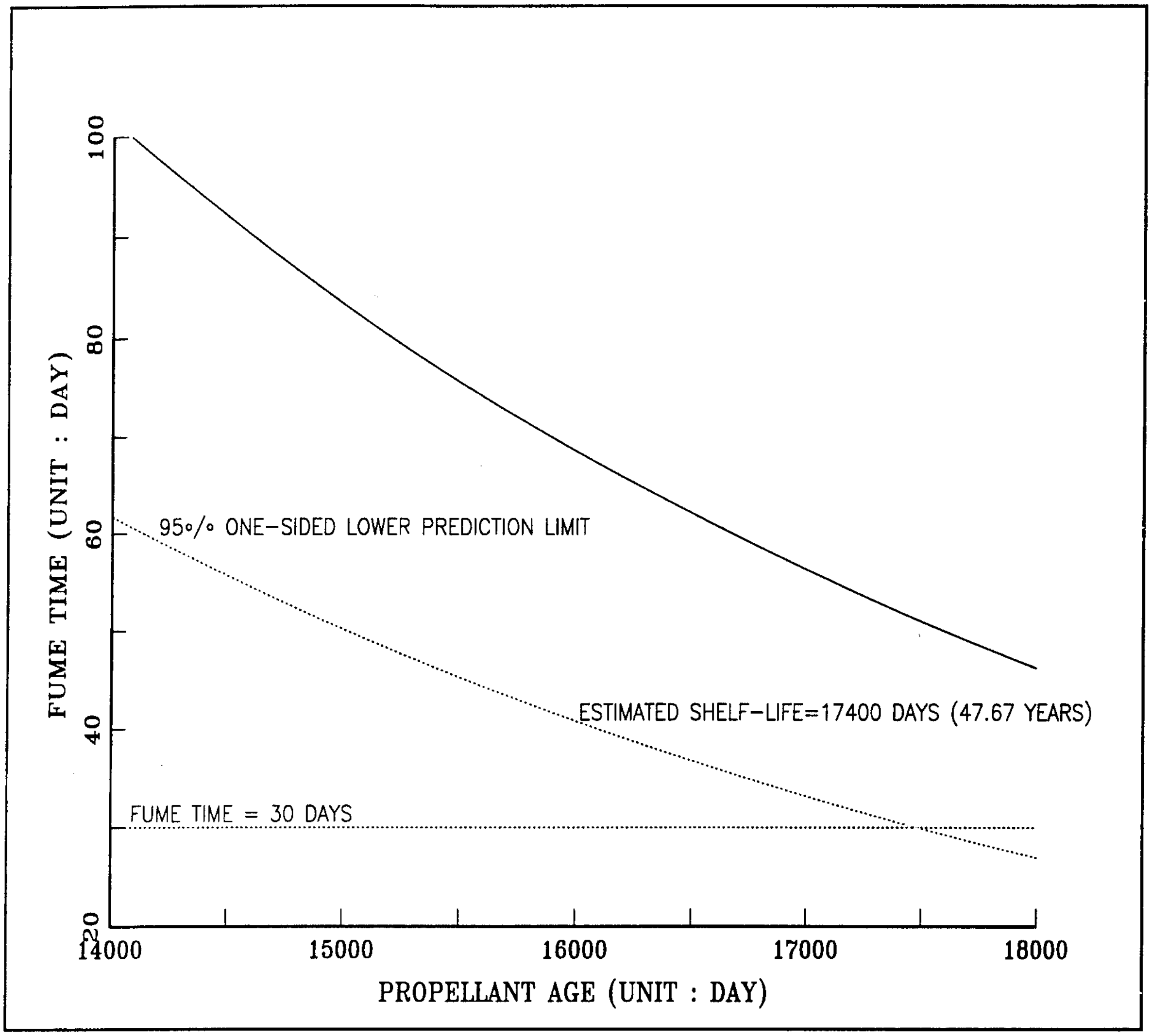

Figure C.11. Group shelf-life estimation based on the linearizing transformation model II.

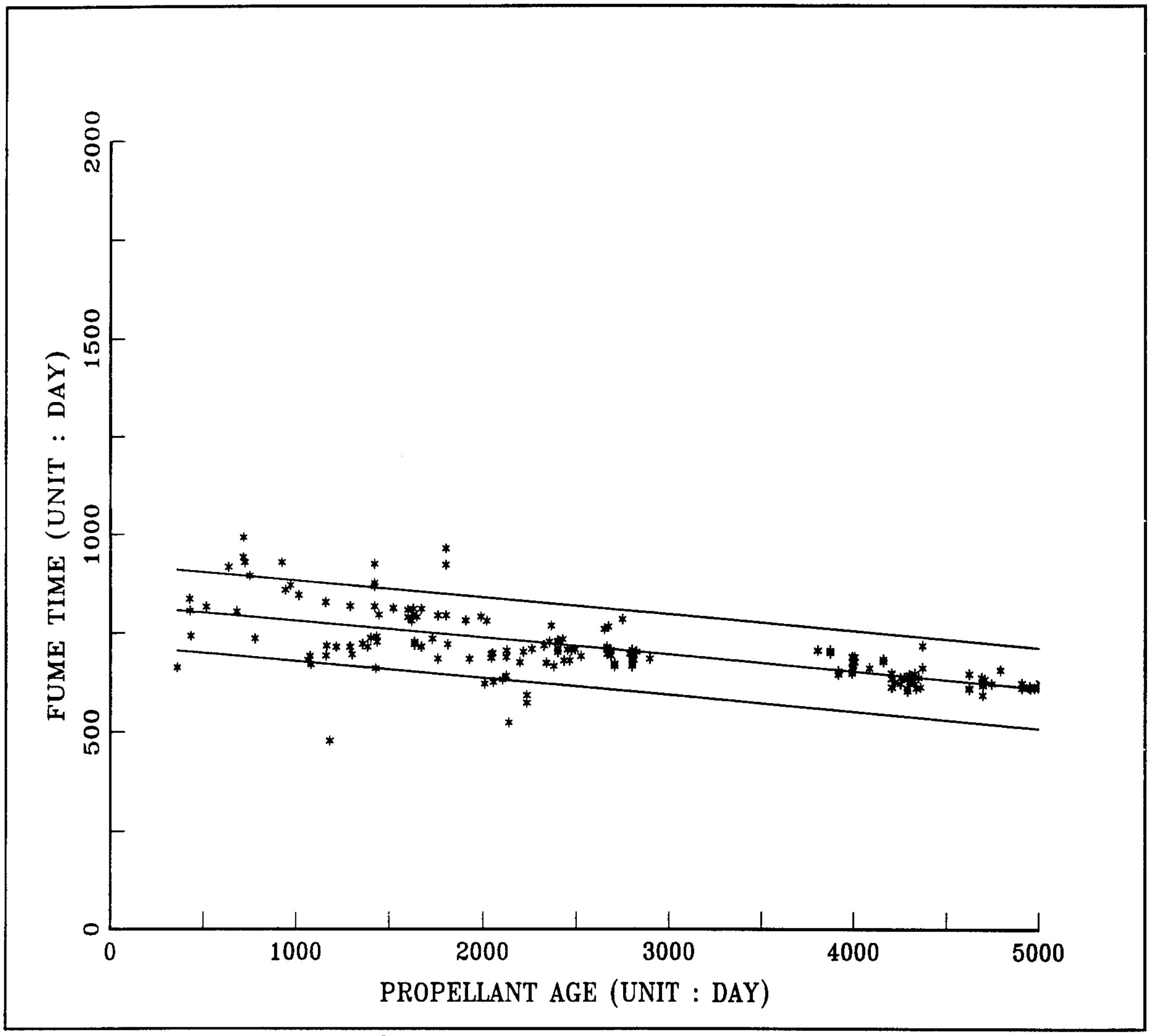

Figure C.12. Fume time vs propellant age based on the fleet return data.

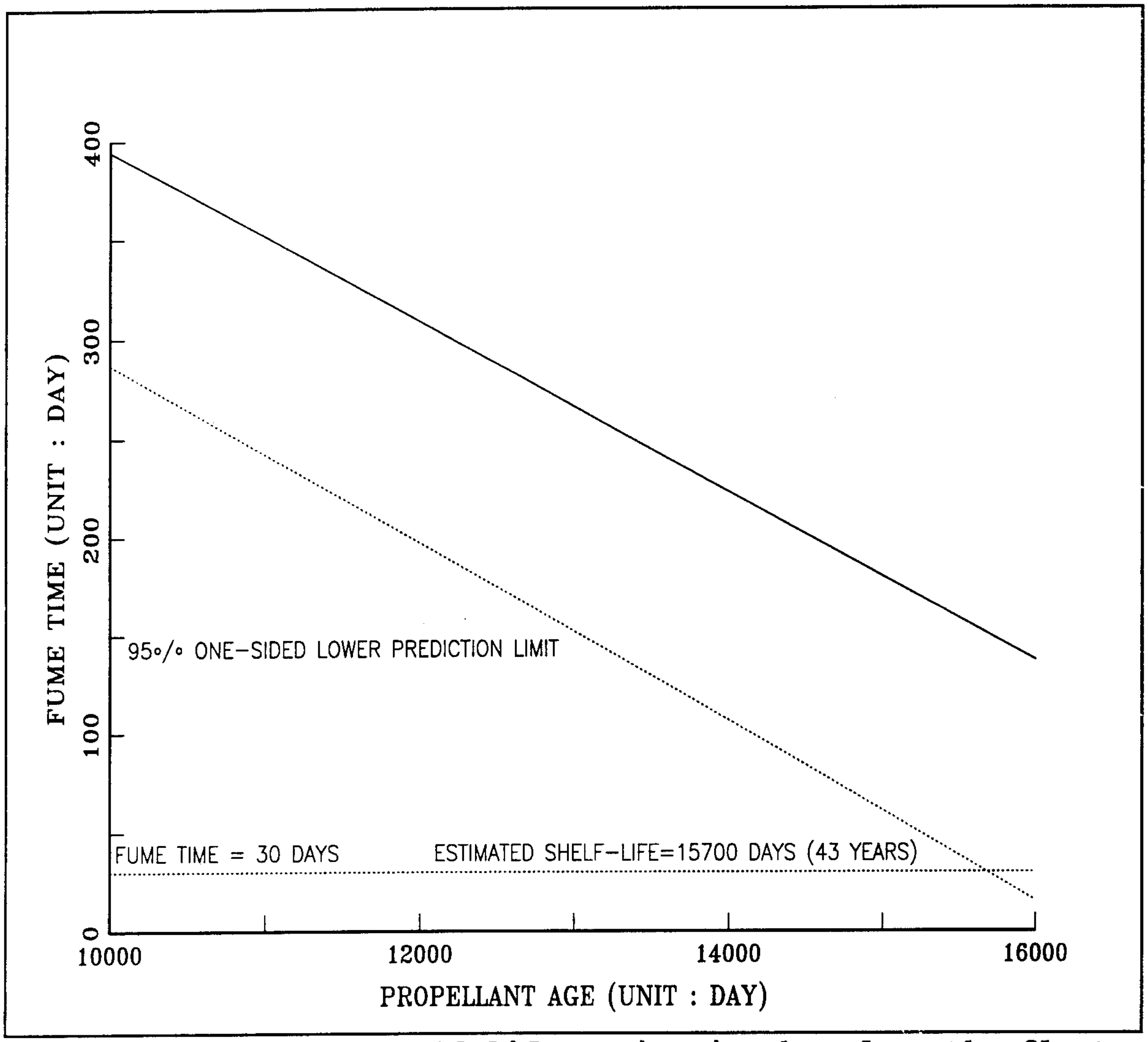

Figure C.13. Group shelf-life estimation based on the fleet return data

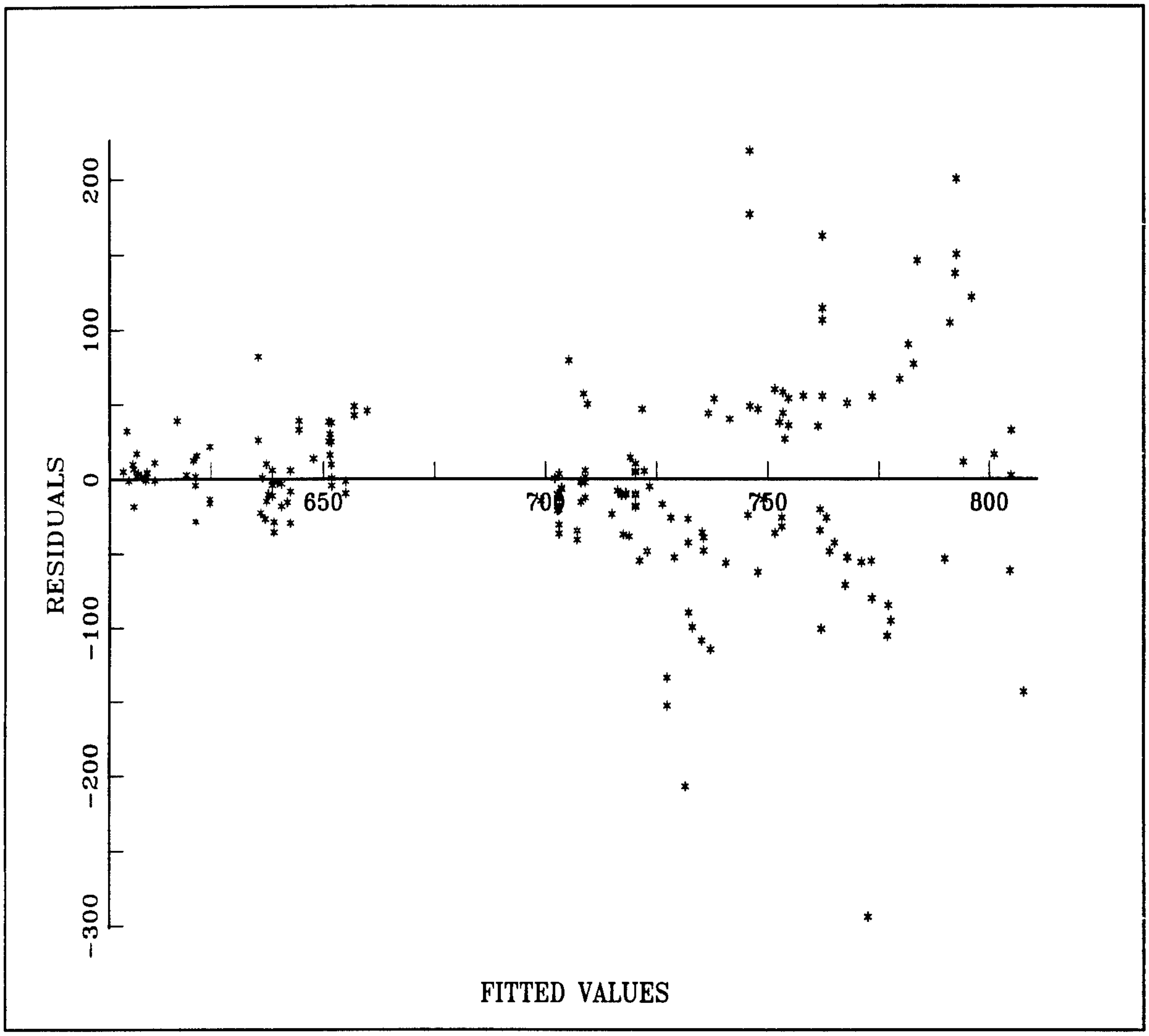

Figure C.14. Residual analysis based on the fleet return data

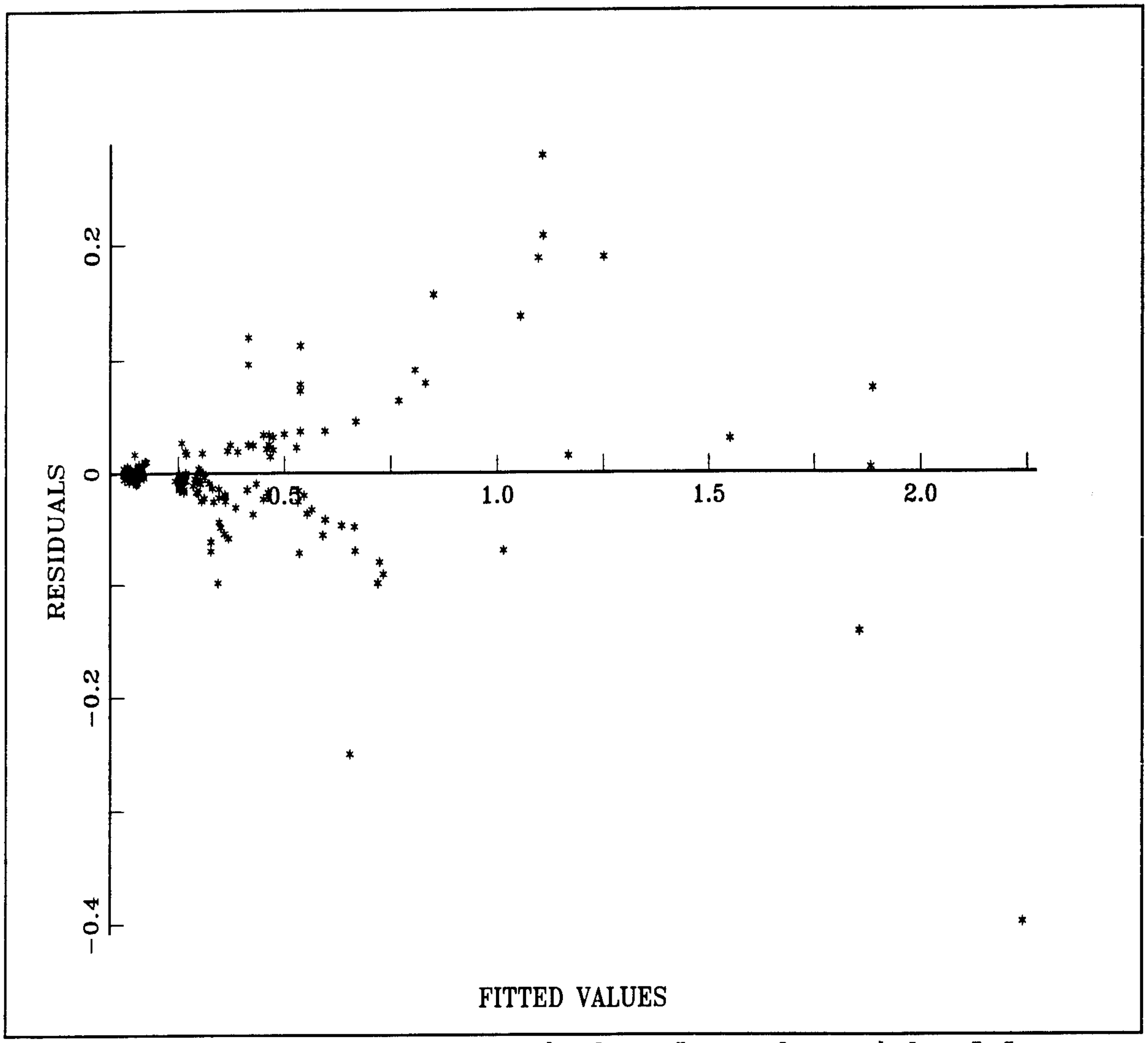

Figure C.15. Residual analysis based on the weighted least square model.

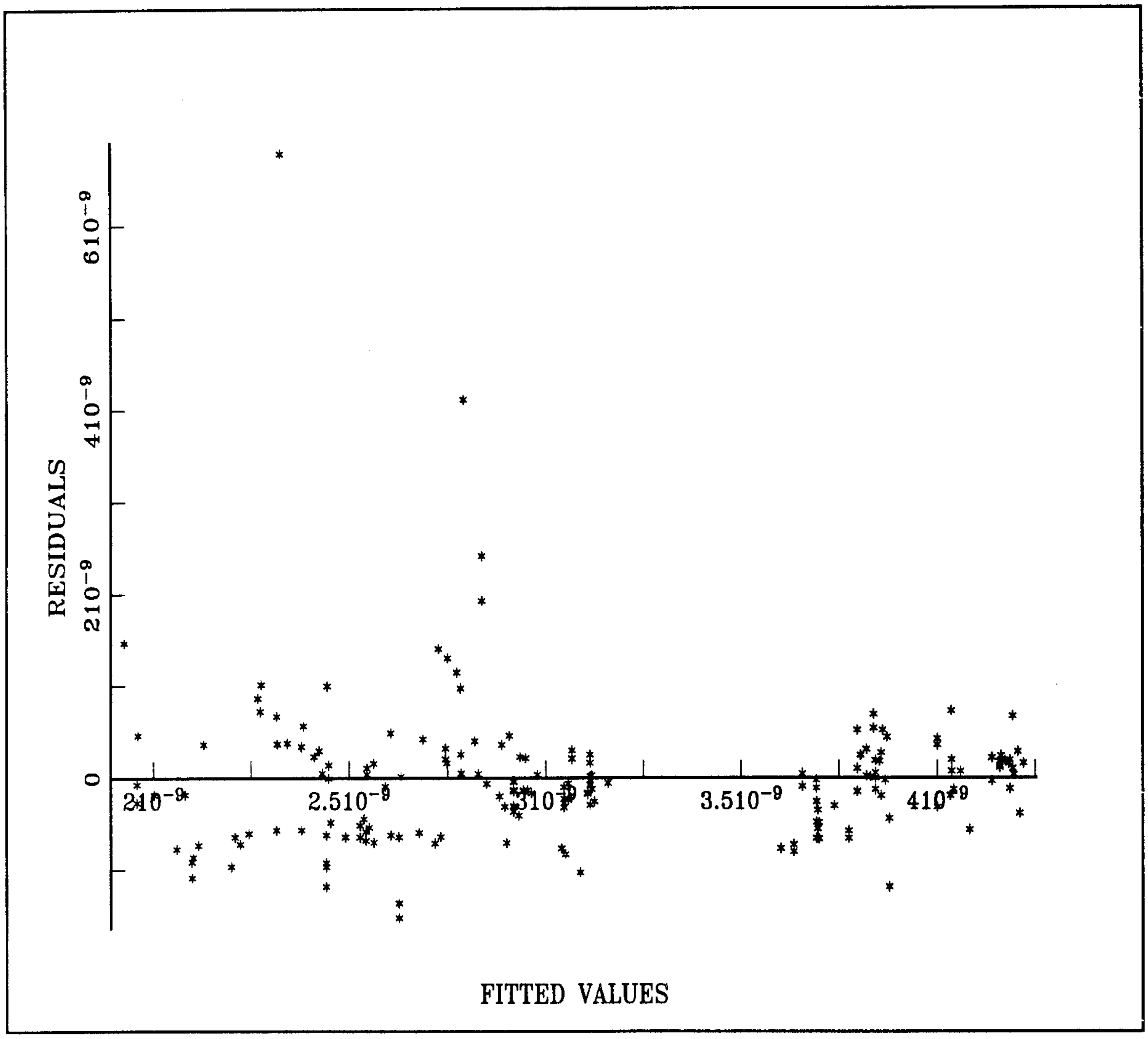

Figure C.16. Residual analysis based on the power transformation model.

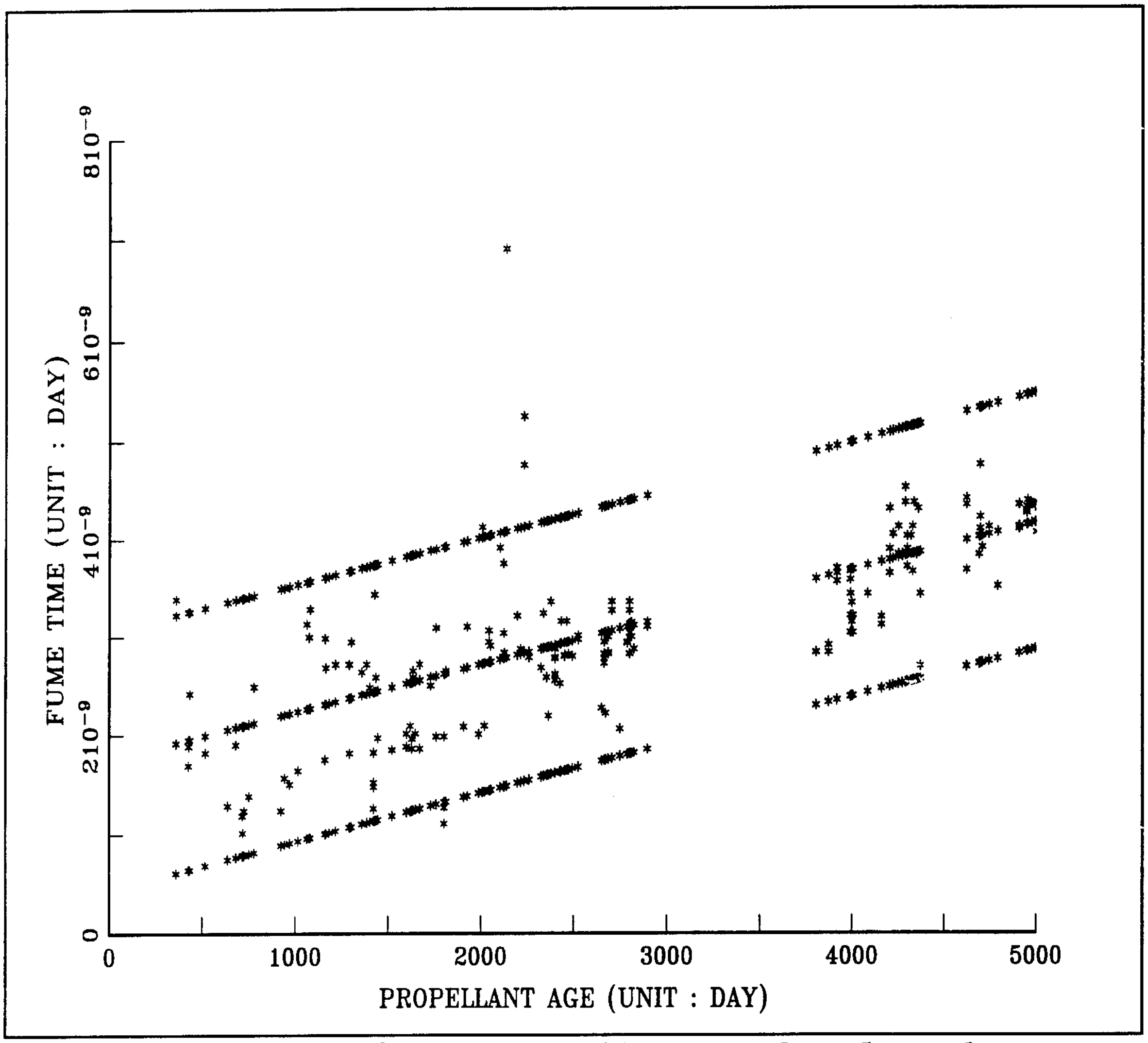

Figure C.17. Fume time vs propellant age based on the power transformation model.

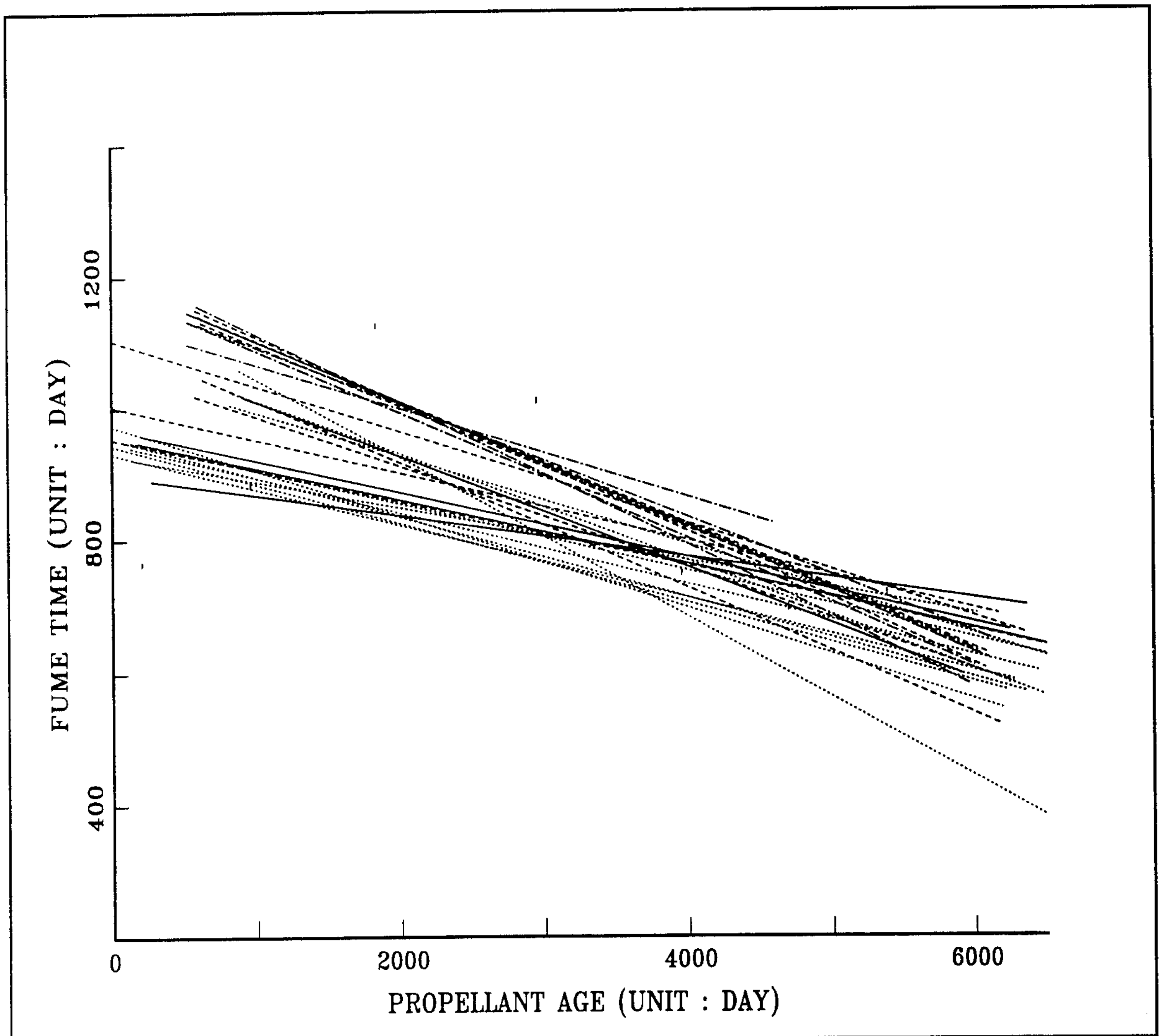

Figure C.18. Fitted within-individual models based on the standard linear regression model I.

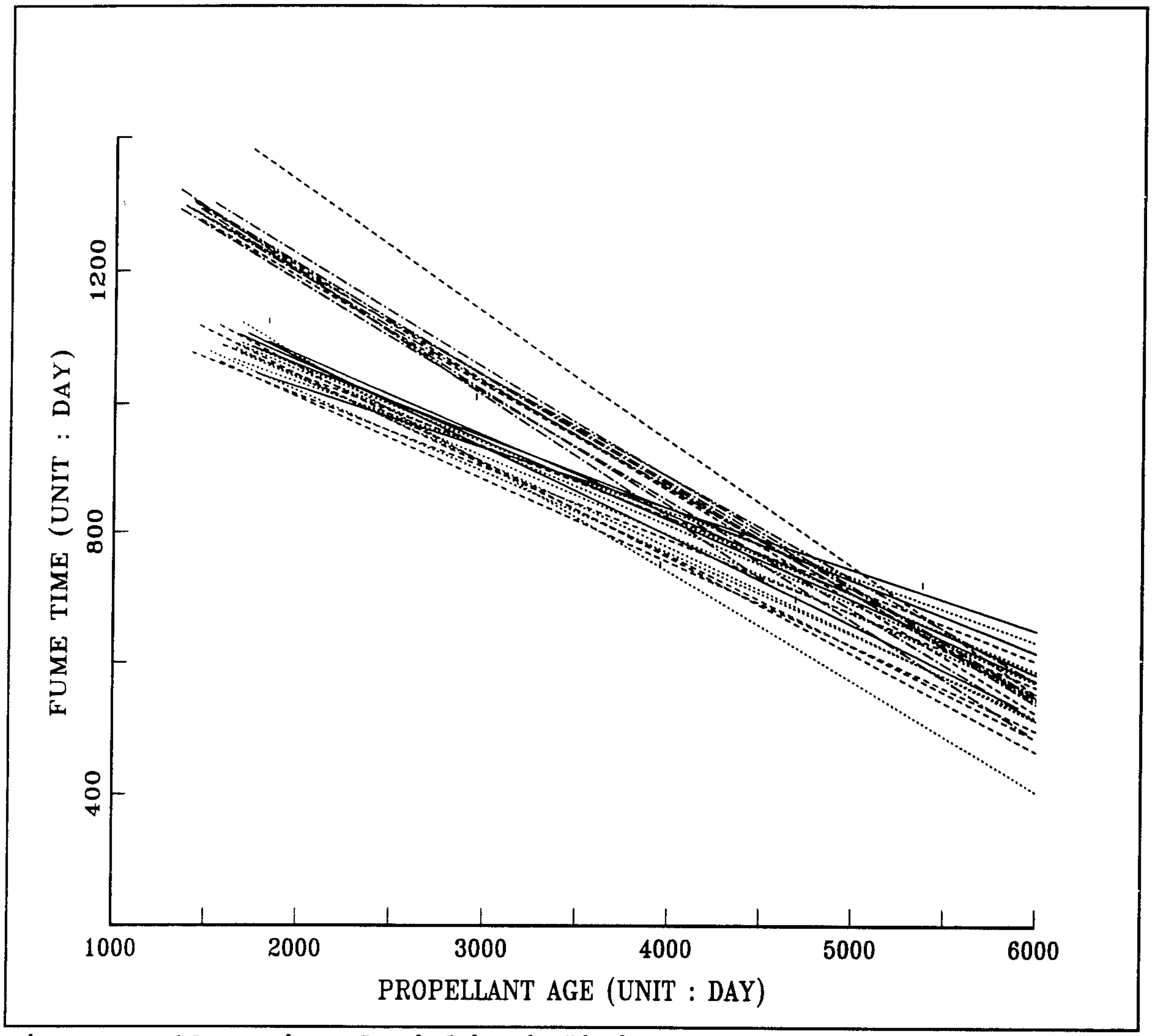

Figure C.19. Fitted within-individual models based on the standard linear regression model II.

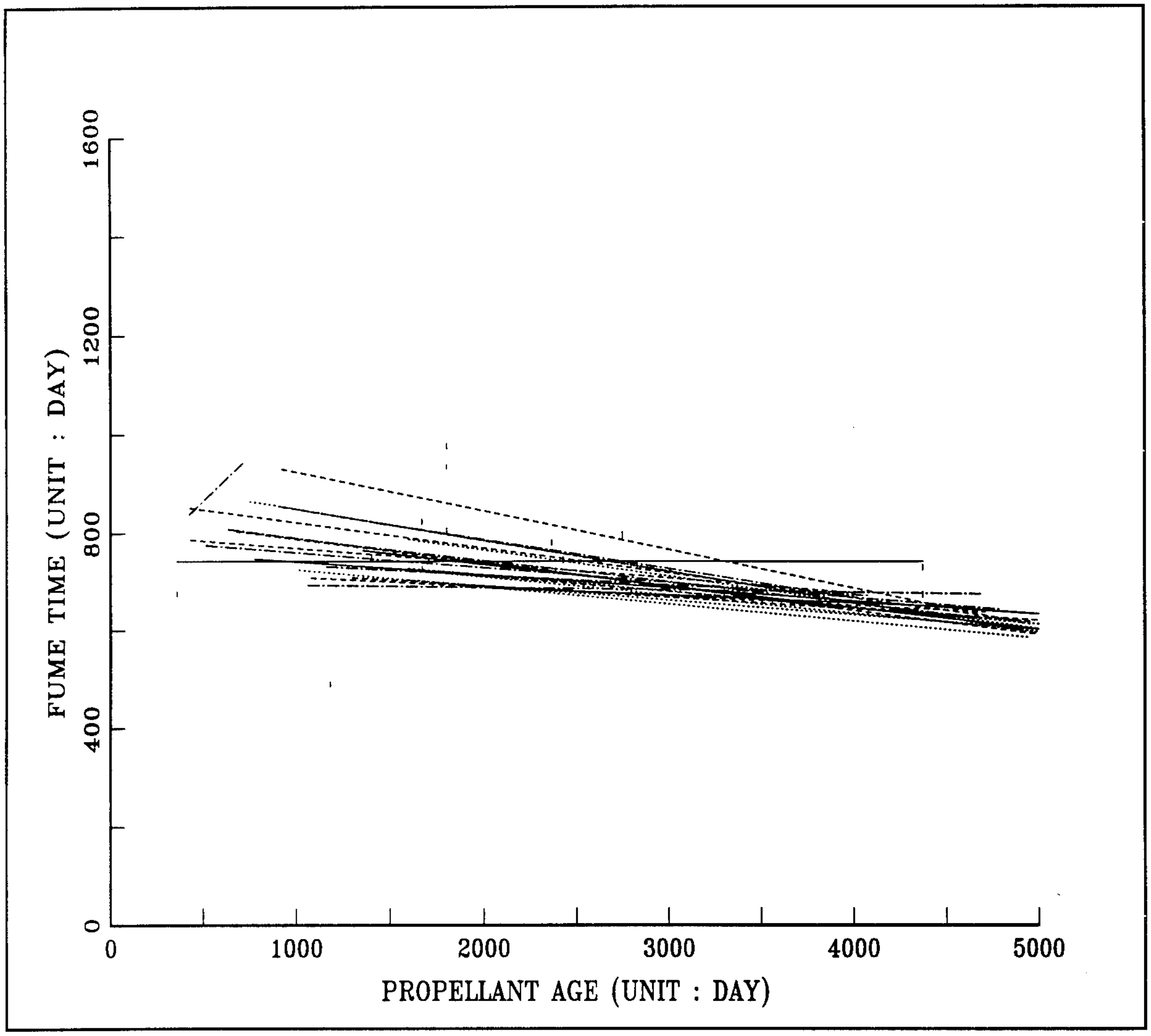

Figure C.20. Fitted within-individual models based on the fleet return data.

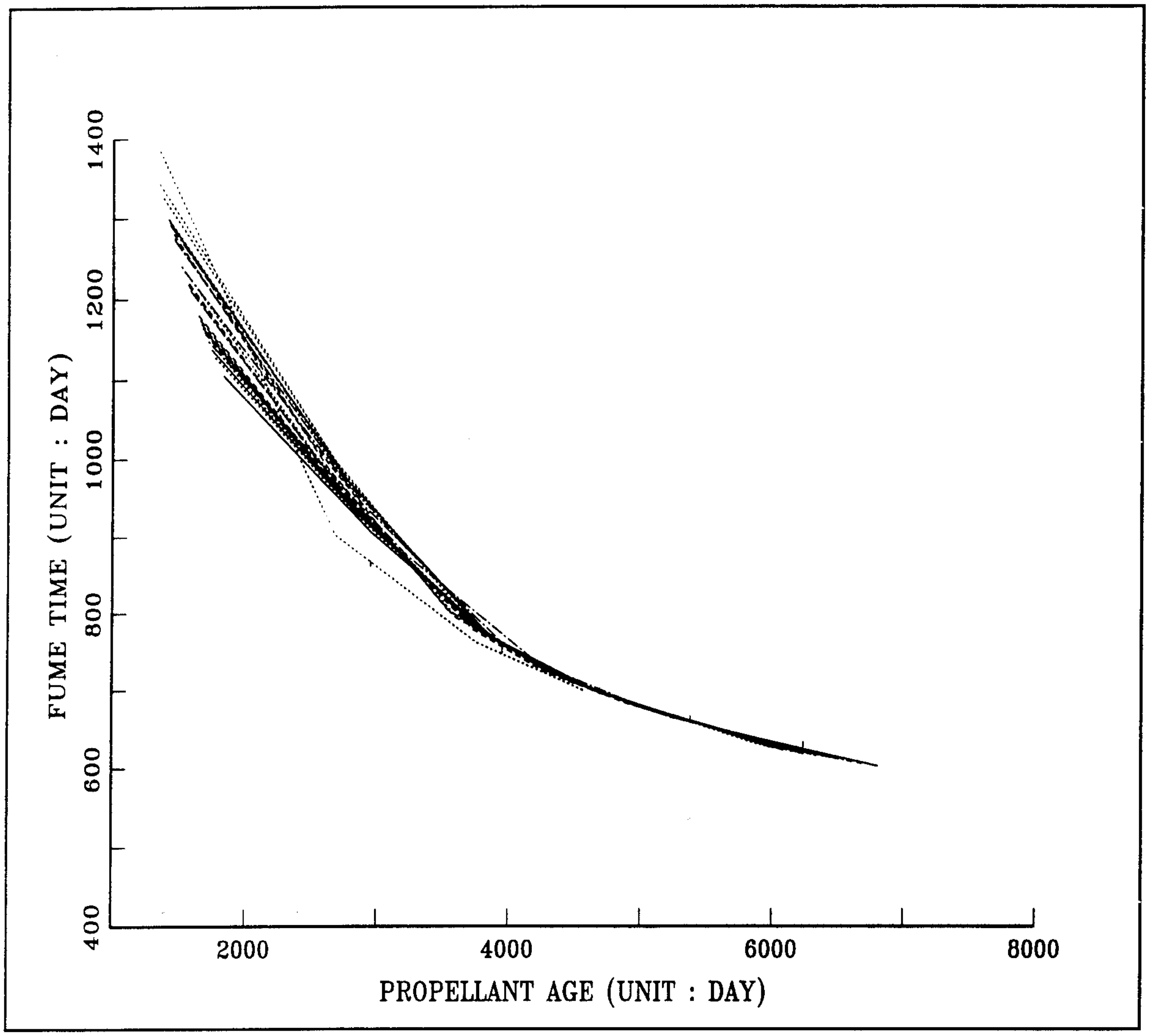

Figure C.21. Fitted within-individual models based on the linearizing transformation model I.

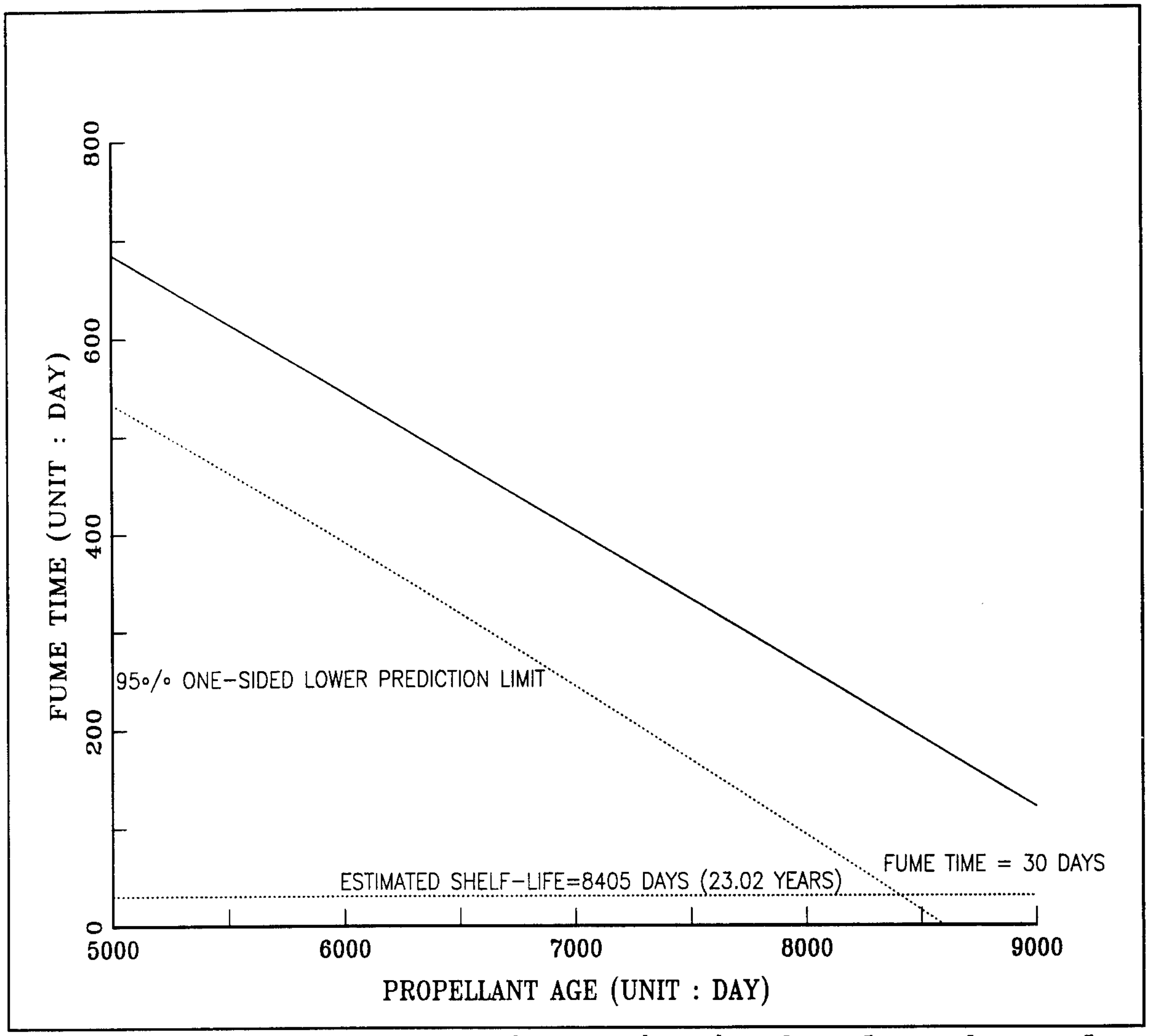

Figure C.22. Group shelf-life estimation based on the random effects regression model.

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